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NAS SAUFLEY FIELD
5090.3a

FINAL SAMPLING AND ANALYSIS PLAN FOR GROUNDWATER AND SOIL SAMPLING AT SITE 5 NAS SAUFLEY FIELD FL

2/1/2011

TETRA TECH

SAP Worksheet #1 -- Title and Approval Page
(UFP-QAPP Manual Section 2.1)

FINAL
SAMPLING AND ANALYSIS PLAN
(Field Sampling Plan and Quality Assurance Project Plan)
September 2010

Groundwater and Soil Sampling
Saufley Field, Site 5

Pensacola, Florida

Prepared for:
Naval Facilities Engineering Command
Southeast
Naval Air Station Jacksonville Building 903
Jacksonville, Florida 32212-0030

Prepared by:
Tetra Tech NUS, Inc.
234 Mall Boulevard
King of Prussia, Pennsylvania 19406-2954

Prepared under:
Comprehensive Long-term Environmental Action Navy
(CLEAN) Contract No. N62470-08-D-1001
Contract Task Order JM26

Review Signatures: T. Johnston 2-25-2011
Tetra Tech NUS, Inc. Quality Assurance Manager
Tom Johnston, PhD

Other Review Signatures: Geoff C.P.
Tetra Tech NUS, Inc. Project Manager
Geoff Pope, P.E.

Approval Signatures: ____________________
NAVFAC Quality Assurance Officer/Chemist

Other Approval Signatures: REED.SARAH.M.1239517036 Digitally signed by REED.SARAH.M.1239517036 DN: c=US, o=U.S. Government, ou=DoD, ou=PKI, ou=USN, cn=REED.SARAH.M.1239517036 Date: 2011.02.25 11:42:34 -05'00'
NAVFAC Remedial Project Manager
Sarah Reed

Other Approval Signatures: David P. Grabka 3/4/11
Florida Department of Environmental Protection
David Grabka

SAP Worksheet #1 -- Title and Approval Page
(UFP-QAPP Manual Section 2.1)

DRAFT
SAMPLING AND ANALYSIS PLAN
(Field Sampling Plan and Quality Assurance Project Plan)
September 2010

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Prepared for:
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Review Signatures: ______________________
Tetra Tech NUS, Inc. Quality Assurance Manager
Tom Johnston, PhD

Other Review Signatures: ______________________
Tetra Tech NUS, Inc. Project Manager
Gerald Walker

Approval Signatures: BOWERS.KENNETH.A.1230092474 Digitally signed by BOWERS.KENNETH.A.1230092474 DN: c=US, o=U.S. Government, ou=DoD, ou=PKI, ou=USN, cn=BOWERS.KENNETH.A.1230092474 Date: 2010.12.15 16:09:33 -05'00'
NAVFAC Quality Assurance Officer/Chemist

Other Approval Signatures: ______________________
NAVFAC Remedial Project Manager
Sarah Reed

Other Approval Signatures: ______________________
Florida Department of Environmental Protection
David Grabka

EXECUTIVE SUMMARY

This Uniform Federal Policy Sampling and Analysis Plan (UFP-SAP) describes the investigation designed to collect groundwater and soil samples from Saufley Field, Site 5, located in Pensacola, Florida. The UFP-SAP was prepared by Tetra Tech NUS, Inc. (Tetra Tech) on behalf of Naval Facilities Engineering Command (NAVFAC) Southeast under Contract Number N62470-08-D-1001, Contract Task Order (CTO) JM26. The field investigation described herein will be conducted to identify and delineate areas of contamination, if present, at Site 5.

Saufley Field is located in Escambia County, between Interstate 10 and Perdido Bay, approximately 5 miles northwest of Pensacola, Florida, in the northwest coastal section of the Florida panhandle. The installation's main complex currently encompasses approximately 866 acres and includes a number of support buildings, a federal prison located south of the airfield, four airstrips, and undeveloped lands. The area currently occupied by Saufley Field included farms and woodlands before it was purchased by the Navy in the 1930s.

Site 5 consists solely of the aviation gasoline (AVGAS) and jet fuel (JP-4) fuel distribution lines connecting the underground storage tank (UST) farm to the flight line and refueling pits, or bowsers, located along the aircraft parking ramp (tarmac). The UST farm enclosure consisted of six 25,000-gallon AVGAS USTs and one 15,000-gallon UST containing JP-4. AVGAS and JP-4 were distributed through 4-inch, 8-inch, and 10-inch diameter steel underground lines connecting the USTs to the flight line bowsers. There were 55 bowsers arrayed in two parallel lines (a south line and a north line) along the aircraft parking ramp.

This UFP-SAP includes collection and analysis of both environmental and background soil and groundwater samples. These samples will be submitted to a Florida Department of Environmental Protection (FDEP)-approved National Environmental Laboratory Accreditation Program (NELAP)-certified laboratory for analysis, as well as other testing to meet the FDEP requirements for Petroleum Storage Tank System Closure Assessments [as defined in Chapter 62-761, Florida Administrative Code (F.A.C.)]. The investigation will require the installation of permanent groundwater sampling points. Depth to groundwater is estimated to be approximately 45 to 50 feet below land surface (bls).

This UFP-SAP was generated for, and complies with, applicable United States (U.S.) Department of the Navy (Navy), U.S. Environmental Protection Agency (USEPA), and FDEP requirements, regulations, guidance, and technical standards. This includes the Department of Defense (DoD), Department of Energy (DOE), and USEPA Interagency Data Quality Task Force (IDQTF) environmental requirements regarding federal facilities. To comply with IDQTF requirements, this UFP-SAP is presented in the format of 37 standard worksheets specified in the Uniform Federal Policy for Quality Assurance Project Plans (UFP-QAPP) guidance documents (USEPA, 2005).

SAP Worksheets

List of Figures

Appendices

ACRONYMS AND ABBREVIATIONS

^{o}C	Degrees Celsius
%D	Percent Difference or Percent Drift
%R	Percent Recovery
%RSD	Percent Relative Standard Deviation
AES	Atomic Emission Spectroscopy
ASP	Alternate Sampling Plan
AST	Aboveground Storage Tank
AVGAS	Aviation Gasoline
BFB	Bromofluorobenzene
bgs	Below Ground Surface
bls	Below Land Surface
BTEX	Benzene, Toluene, Ethylbenzene, and Total Xylenes
CA	Corrective Action
CAS	Chemical Abstracts Service
CCB	Continuing Calibration Blank
CCC	Continuing Calibration Compound
CCV	Continuing Calibration Verification
CFR	Code of Federal Regulations
CLEAN	Comprehensive Long-term Environmental Action Navy
CLP	Contract Laboratory Program
CSM	Conceptual Site Model
CTL	Contaminant Target Level
CTO	Contract Task Order
DFTPP	Decafluorotriphenylphosphine
DI	Deionized
DO	Dissolved Oxygen
DoD	Department of Defense
DoD QSM	Department of Defense Quality Systems Manual for Environmental Laboratories
DOE	Department of Energy
DOT	Department of Transportation
DPT	Direct-Push Technology
DQI	Data Quality Indicator
DVM	Data Validation Manager
ECD	Electron Capture Detector
EDB	Ethylene Dibromide
EDD	Electronic Data Deliverable
ELAP	Environmental Laboratory Accreditation Program

Empirical	Empirical Laboratories, LLC
Ext.	Extension
F.A.C.	Florida Administrative Code
FDEP	Florida Department of Environmental Protection
FDOH	Florida Department of Health
FID	Flame Ionization Detector
FOL	Field Operations Leader
FTMR	Field Task Modification Request
GC/ECD	Gas Chromatography Electron Capture Detector
GC/FID	Gas Chromatography Flame Ionization Detector
GC/MS	Gas Chromatograph/Mass Spectrometer
GCTL	Groundwater Cleanup Target Level
GPS	Global Positioning System
HASP	Health and Safety Plan
HCl	Hydrochloric Acid
HNO_3	Nitric Acid
HSM	Health and Safety Manager
ICAL	Initial Calibration
ICB	Initial Calibration Blank
ICP	Inductively Coupled Plasma
ICP-AES	Inductively Coupled Plasma - Atomic Emission Spectroscopy
ICS	Interference Check Standard
ICV	Initial Calibration Verification
ID	Inner Diameter
IDQTF	Intergovernmental Data Quality Task Force
IDW	Investigation-Derived Waste
IRP	Installation Restoration Program
IS	Internal Standard
L	Liter
LCS	Laboratory Control Sample
LCSD	Laboratory Control Sample Duplicate
LIMS	Laboratory Information Management System
LNAPL	Light Non-Aqueous Phase Liquid
LOD	Limit of Detection
LOQ	Limit of Quantitation
MDL	Method Detection Limit
mg/kg	Milligrams per Kilogram
mL	Milliliter
MPC	Measurement Performance Criterion

MS	Matrix Spike
MSD	Matrix Spike Duplicate
msl	Mean Sea Level
MTBE	Methyl Tert-Butyl Ether
NA	Not Applicable
NAAS	Naval Auxiliary Air Station
NAS	Naval Air Station
NAVD	North America Vertical Datum
NAVFAC	Naval Facilities Engineering Command
Navy	United States Department of the Navy
ND	Non Detect
NELAP	National Environmental Laboratory Accreditation Program
NETPDTC	Naval Education and Training Professional Development and Technical Center
NETPMSA	Naval Educational and Training Program Management Support Activity
NEESA	Naval Energy and Environmental Support Activity
NFA	No Further Action
NGVD	National Geodetic Vertical Datum
NIRIS	Naval Installation Restoration Information Solution
NSF	National Sanitation Foundation
NTTC	Navy Technical Training Center
NTU	Nephelometric Turbidity Unit
OSHA	Occupational Health and Safety Administration
PAH	Polycyclic Aromatic Hydrocarbon
PAL	Project Action Limit
PM	Project Manager
POC	Point of Contact
ppm	Parts Per Million
PPL	Priority Pollutant List
PQL	Practical Quantitation Limit
PQO	Project Quality Objective
PVC	Polyvinyl Chloride
PWC	Public Works Center
PW	Potable Water
QA	Quality Assurance
QAM	Quality Assurance Manager
QAO	Quality Assurance Officer
QC	Quality Control
r	Linear Regression Correlation Coefficient
r^2	Coefficient of Determination

RAP	Remedial Action Plan
RF	Response Factor
RPD	Relative Percent Difference
RPM	Remedial Project Manager
RRT	Relative Retention Time
RT	Retention Time
SAP	Sampling and Analysis Plan
SAR	Site Assessment Report
SCTL	Soil Cleanup Target Limit
SDG	Sample Delivery Group
SOP	Standard Operating Procedure
SPCC	System Performance Check Compound
SSO	Site Safety Officer
SunLabs	SunLabs, Inc. – Central Laboratory
TBD	To Be Determined
Tetra Tech	Tetra Tech NUS, Inc.
TPH	Total Petroleum Hydrocarbons
TPHCWG	Total Petroleum Hydrocarbons Criteria Working Group
TRPH	Total Recoverable Petroleum Hydrocarbons
UFP-QAPP	Uniform Federal Policy for Quality Assurance Project Plans
UFP-SAP	Uniform Federal Policy Sampling and Analysis Plan
μg/L	micrograms per liter
U.S.	United States
USEPA	United States Environmental Protection Agency
UST	Underground Storage Tank
UVF	Ultraviolet Fluorescence
VOC	Volatile Organic Compound
VOH	Volatile Organic Halocarbon

SAP Worksheet #2 -- SAP Identifying Information

(UFP-QAPP Manual Section 2.2.4)

Site Name/Number:	**Saufley Field, Pensacola, Florida**
Operable Unit:	**Site 5**
Contractor Name:	**Tetra Tech NUS, Inc. (Tetra Tech)**
Contract Number:	**N62470-08-D-1001**
Contract Title:	**Navy Comprehensive Long-term Environmental Action Navy (CLEAN)**
Work Assignment Number:	**Contract Task Order (CTO) JM26**

1. This Sampling and Analysis Plan (SAP) was prepared in accordance with the requirements of the United States Environmental Protection Agency (USEPA) *Uniform Federal Policy for Quality Assurance Project Plans* (UFP-QAPP) (USEPA, 2005) and the USEPA *Guidance for Quality Assurance Project Plans, QA-G5, QAMS* (USEPA, 2002a).

2. Identify regulatory program: Florida Department of Environmental Protection (FDEP) Underground Storage Tank (UST) Program.

3. This SAP is a project-specific SAP.

4. List dates of scoping sessions that were held:

Scoping Session	Date
Pre-Data Quality Objectives (DQO) Scoping Session	April 27, 2010
DQO Conference Call	June 2, 2010

5. List dates and titles of any SAP documents written for previous site work that are relevant to the current investigation.

Title	Date
Preliminary Assessment Report, Saufley Field, Escambia County, Florida	1992
Alternative Sampling Plan for Site 5, Saufley Field, Escambia County, Florida	June 2010
NAVFAC DWG #s 5134920 thru 5134925	1985

6. List organizational partners (stakeholders) and connection with lead organization:
FDEP (regulatory oversight) and NAS Pensacola (property owner).

7. Lead organization: NAVFAC Southeast

8. If any required SAP elements or required information are not applicable to the project or are provided elsewhere, then note the omitted SAP elements and provide an explanation for their exclusion below:
Not Applicable (NA), as there are no exclusions.

SAP Worksheet #3 -- Distribution List
(UFP-QAPP Manual Section 2.3.1)

Name of SAP Recipient	Title/Role	Organization	Telephone Number	E-Mail Address or Mailing Address
Sarah Reed	Navy Remedial Project Manager (RPM)/ Manages Project Activities for the Navy	NAVFAC SE Integrated Product Team South Central Code OPZE3 Building 903 Jacksonville, FL 32212-0030	904-452-6290	w_gates@bellsouth.net
Greg Campbell	Installation Restoration Program (IRP) Manager/ Naval Air Station (NAS) Pensacola and Saufley Field Point of Contact (POC)	NAS Pensacola Public Works Center (PWC) 310 John Tower Road Pensacola, FL 32508-5000	850-452-3131 Extension (Ext.) 3007	gregory.campbell@navy.mil
To Be Determined (TBD)	NAVFAC Quality Assurance Officer (QAO)/ Navy Chemist	TBD	TBD	TBD
TBD	Head of Reference Desk (Saufley Field Administrative Record)	TBD	TBD	TBD
Bonnie Capito	Administrative Record/ Librarian	NAVFAC Atlantic	757-322-4785	bonnie.capito@navy.mil
David Grabka	FDEP Project Manager (PM)/ Provides Regulator Input	Florida Department of Environmental Protection 2600 Blair Stone Road, MS 4535 Tallahassee, FL 32399-2400	850-245-8997	david.grabka@dep.state.fl.us
John Trepanowski (copy of cover letter only)	Tetra Tech Program Manager / Manages Navy Initiatives	Tetra Tech 234 Mall Boulevard Suite 260 King of Prussia, PA 19406	610-382-1532	john.trepanowski@tetratech.com

Name of SAP Recipient	Title/Role	Organization	Telephone Number	E-Mail Address or Mailing Address
Garth Glenn (copy of cover letter only)	Tetra Tech Deputy Program Manager/ Manages Program Activities	Tetra Tech 5700 Lake Wright Drive Suite 309 Norfolk, VA 23502	757-461-3926	garth.glenn@tetratech.com
Geoff Pope	PM/ Manages Project Activities	Tetra Tech 65 Union Avenue Suite 300 Memphis, TN 38103	901-523-9500	geoff.pope@tetratech.com
Jim Coffman	Project Geophysicist/ Provides Buried Utility Clearances	Tetra Tech 661 Andersen Drive Foster Plaza 7 Pittsburgh, PA 15220	412-921-8244	james.coffman@tetratech.com
Yarissa Martinez	Field Operations Leader (FOL)/ Site Safety Officer (SSO)/ Manages Field Operations and Site Safety Issues	Tetra Tech 1558 Village Square Boulevard Suite 2 Tallahassee, FL 32309	850-385-9899 Ext. 1355	yarissa.martinez@tetratech.com
Matt Soltis [Health and Safety Plan (HASP) only]	Health and Safety Manager (HSM)/ Manages Corporate Health and Safety Program	Tetra Tech 661 Andersen Drive Foster Plaza 7 Pittsburgh, PA 15220	412-921-8912	matt.soltis@tetratech.com
Tom Johnston (electronic copy only)	Quality Assurance Manager (QAM)/ Manages Corporate Quality Assurance (QA) Program and Implementation	Tetra Tech 661 Andersen Drive Foster Plaza 7 Pittsburgh, PA 15220	412-921-8615	tom.johnston@tetratech.com
Matt Kraus (electronic copy only)	Project Chemist/ Provides Coordination with Laboratory	Tetra Tech 661 Andersen Drive Foster Plaza 7 Pittsburgh, PA 15220	412-921-8729	matt.kraus@tetratech.com

Name of SAP Recipient	Title/Role	Organization	Telephone Number	E-Mail Address or Mailing Address
Joseph Samchuck (electronic copy only)	Tetra Tech Data Validation Manager (DVM)/ Manages Data Validation	Tetra Tech 661 Andersen Drive Foster Plaza 7 Pittsburgh, PA 15220	412-921-8510	joseph.samchuck@tetratech.com
Lee Leck (electronic copy only)	Tetra Tech Data Manager/ Manages Databases	Tetra Tech 661 Andersen Drive Foster Plaza 7 Pittsburgh, PA 15220	412-921-8856	lee.leck@tetratech.com
Kim Kostzer (electronic copy only)	Laboratory PM/ Representative for Laboratory and Analytical Issues	Empirical Laboratories, LLC (Empirical) 621 Mainstream Drive, Suite 270 Nashville, TN 37228	615-345-1115	kkostzer@empirlabs.com
Lori Palmer (electronic copy only)	Laboratory PM/ Representative for Laboratory and Analytical Issues	SunLabs Inc. – Central Laboratory (SunLabs) 5460 Beaumont Center Boulevard Tampa, FL 33634	813-881-9401	lpalmer@sunlabsinc.com
TBD (electronic copy only)	Drilling Subcontractor PM/ Provides Direct-Push Technology (DPT) Services	TBD	TBD	TBD
TBD (electronic copy only)	Well Installation PM/ Provides Well Installation Services	TBD	TBD	TBD

Each person in this table will be responsible for distributing copies of this SAP to appropriate personnel within their organization. For example, the Tetra Tech PM will be responsible for distributing copies of this SAP to all Tetra Tech personnel listed in Worksheet #4 (Project Personnel Sign-Off Sheet).

SAP Worksheet #4 -- Project Personnel Sign-Off Sheet
(UFP-QAPP Manual Section 2.3.2)

Certification that project personnel have read the text will be obtained by one of the following methods as applicable:

1. In the case of regulatory agency personnel with oversight authority, approval letters or e-mails will constitute verification that applicable sections of the SAP have been reviewed. Copies of regulatory agency approval letters / e-mails will be retained in the project files as project records (see Worksheet #29).

2. E-mails will be sent to the listed Navy, Tetra Tech, and subcontractor project personnel whom will be requested to verify by e-mail that they have read the applicable SAP / sections and the date on which they were reviewed. Copies of the verification e-mail will be included in the project files (see Worksheet #29).

A copy of the signed Worksheet #4 will be retained in the project files and identified as a project document in Worksheet #29.

Key personnel will be instructed to read the SAP prior to attending an internal site-specific kick-off meeting for field activities. The Tetra Tech PM will track when the reviews have been completed, obtain signatures, and ensure that the completed sign-off sheet is included in the central project file.

Name[1]	Organization/Title/Role	Telephone Number	Signature/E-Mail Receipt	SAP Section Reviewed	Date SAP Read
Navy and Regulator Project Team Personnel					
Sarah Reed	Navy/ RPM/ Manages Project Activities for the Navy	904-452-6290	See Worksheet #1 for signature	All	
Greg Campbell	Navy/ IRP Manager/ Saufley Field POC	850-452-3131 Ext. 3007		All	
David Grabka	FDEP/ PM/ Provides Regulator Input	850-245-8997	See Worksheet #1 for signature	All	
Tetra Tech Project Team Personnel					
Geoff Pope	Tetra Tech/ PM/ Manages Project Activities	901-523-9500	See Worksheet #1 for signature	All	
Yarissa Martinez	Tetra Tech/ FOL/SSO Manages Field Operations and Site Safety Issues	850-385-9899 Ext. 1355		All	

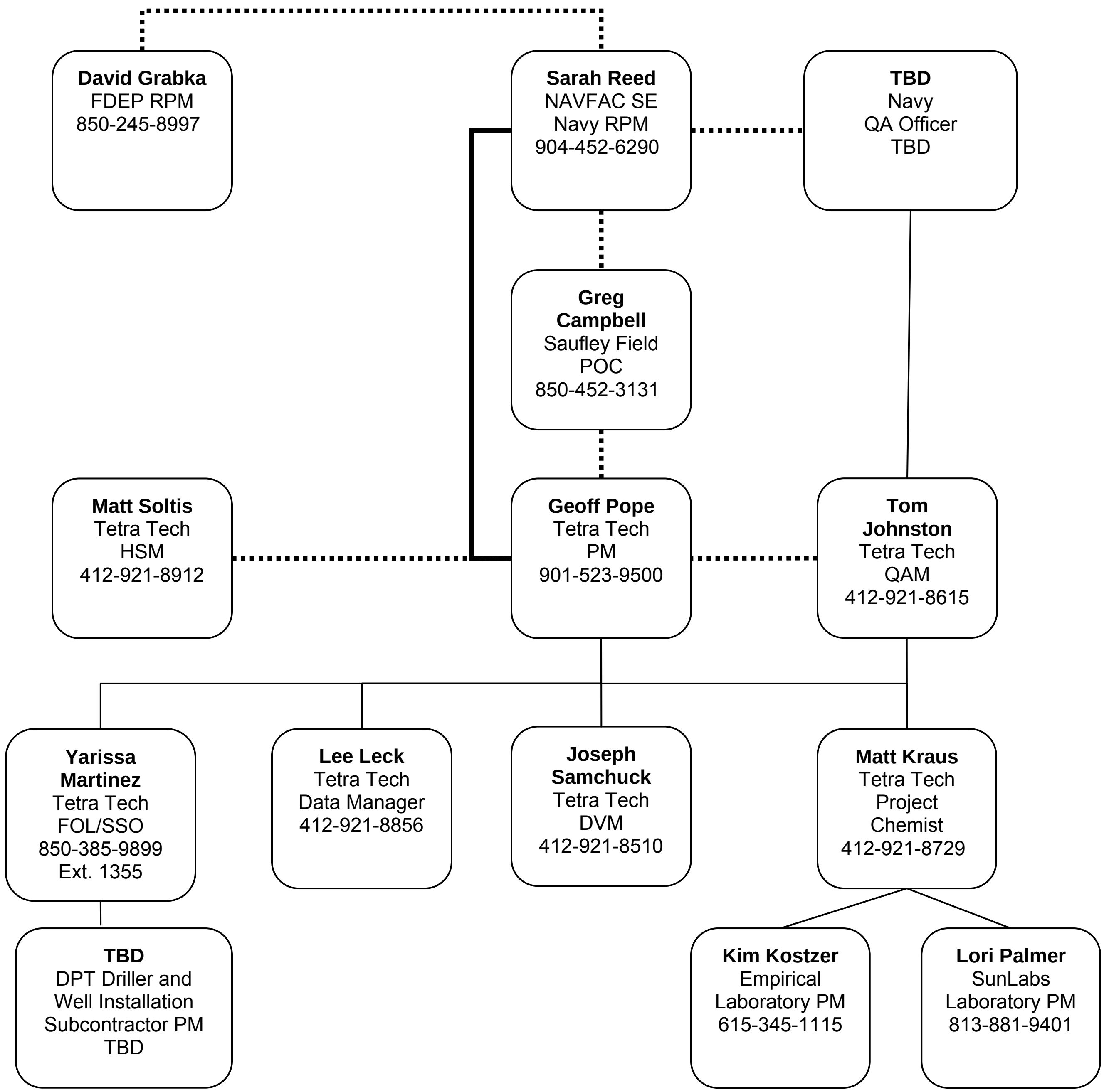
Name[1]	Organization/Title/Role	Telephone Number	Signature/E-Mail Receipt	SAP Section Reviewed	Date SAP Read
Tom Johnston	Tetra Tech/ QAM/ Manages NAVFAC SE Contract QA Program and Implementation	412-921-8615	See Worksheet #1 for signature	All	
Matt Kraus	Tetra Tech/ Project Chemist/ Provides Coordination with Laboratory	412-921-8729		All	
Jim Coffman	Tetra Tech/ Project Geophysicist/ Provides Buried Utility Clearances	412-921-8244		Worksheets #10 and #11	
Matt Soltis	Tetra Tech/ HSM/ Manages Corporate Health and Safety Program	412-921-8912	See HASP for signature	HASP	
Joseph Samchuck	Tetra Tech/ DVM/ Manages Data Validation	412-921-8510		Worksheets #12, #14, #15, #19, #20, #23-28, #30, and #34-37	
Lee Leck	Tetra Tech/ Data Manager/ Manages Databases	412-921-8856		Worksheets #12, #14, #15, #19, #20, #23-28, #30, and #34-37	
Subcontractor Personnel					
Kim Kostzer	Empirical/ Laboratory PM/ Representative for Laboratory and Analytical Issues	615-345-1115		Worksheets #6, #12, #14, #15, #19, #23-28, #30, and #34-36	
Lori Palmer	SunLabs/ Laboratory PM/ Representative for Laboratory and Analytical Issues	813-881-9401		Worksheets #6, #12, #14, #15, #19, #23-28, #30, and #34-36	
TBD	TBD/ Subcontractor PM/ Provides DPT Services	TBD		Worksheets #6, #14, #17, and Figures	

Name[1]	Organization/Title/Role	Telephone Number	Signature/E-Mail Receipt	SAP Section Reviewed	Date SAP Read
TBD	TBD/ Subcontractor PM/ Provides Well Installation Services	TBD		Worksheets #6, #14, #17, and Figures	

1 - Persons listed on this worksheet will be responsible for distributing the SAP to the appropriate people within their organization.

SAP Worksheet #5 -- Project Organizational Chart
(UFP-QAPP Manual Section 2.4.1)

Lines of Authority
Lines of Communication



SAP Worksheet #6 -- Communication Pathways
(UFP-QAPP Manual Section 2.4.2)

Communication Drivers	Responsible Person Affiliation	Name	Phone Number and/or E-Mail	Procedure
SAP amendments	Tetra Tech FOL Tetra Tech PM Navy RPM	Yarissa Martinez Geoff Pope Sarah Reed	850-385-9899 Ext. 1355 901-523-9500 904-452-6290	The Tetra Tech FOL will verbally inform the Tetra Tech PM within 24 hours of realizing a need for an amendment. The Tetra Tech PM will document the proposed changes via a Field Task Modification Request (FTMR) form within five days and send the Navy RPM a concurrence letter within seven days of identifying the need for change for review and approval. The Navy RPM will sign the letter within 5 days of receipt, if approved. The Navy RPM will notify the regulators of changes to the SAP. The Tetra Tech PM will send scope changes to the Project Team via e-mail within one business day.
Schedule changes	Tetra Tech PM Navy RPM Saufley Field POC	Geoff Pope Sarah Reed Greg Campbell	901-523-9500 904-452-6290 850-452-3131, Extension (Ext.) 3007	The Tetra Tech PM will verbally inform the Navy RPM and the Saufley Field POC on the day that schedule change is known and document via a schedule concurrence letter within seven days or prior to the first affected deliverable date.

Communication Drivers	Responsible Person Affiliation	Name	Phone Number and/or E-Mail	Procedure
Field issues that require changes in scope or implementation of field work	Tetra Tech FOL Navy RPM Tetra Tech PM	Yarissa Martinez Sarah Reed Geoff Pope	850-385-9899 Ext. 1355 904-452-6290 901-523-9500	The Tetra Tech FOL will inform the Tetra Tech PM verbally the day the issue is realized. The Tetra Tech PM will inform the Navy RPM of the issue (verbally or via e-mail) within one day of the Tetra Tech FOL's notification. Tetra Tech PM will also send a concurrence letter to the Navy RPM within seven days, if project scope is affected. The Navy RPM will sign the letter within five days of receipt, if scope change is warranted. The scope change is to be implemented before further work is executed. The Tetra Tech PM will document the change via an FTMR form within two days of identifying the need for change and will obtain required approvals within five days of initiating the form. The Tetra Tech PM will place the form in the project file, with signatures as determined by the Tetra Tech PM.
Stop work recommendations, for example, to protect workers from unsafe conditions/situations or to prevent a degradation in quality of work	Tetra Tech FOL Tetra Tech PM Tetra Tech QAM Navy RPM Saufley Field POC	Yarissa Martinez Geoff Pope Tom Johnston Sarah Reed Greg Campbell	850-385-9899 Ext. 1355 901-523-9500 412-921-8615 904-452-6290 850-452-3131, Extension (Ext.) 3007	If Tetra Tech is the responsible party for a stop work command, the Tetra Tech FOL will inform onsite personnel, subcontractor(s), the Saufley Field POC, and the identified Project Team members within one hour (verbally or by e-mail). If a subcontractor is the responsible party, the subcontractor PM must inform the Tetra Tech FOL within 15 minutes, and the Tetra Tech FOL will then follow the procedure listed above.
Field data quality issues	Tetra Tech FOL Tetra Tech PM	Yarissa Martinez Geoff Pope	850-385-9899 Ext. 1355 901-523-9500	The Tetra Tech FOL will inform the Tetra Tech PM verbally or by e-mail on the same day that a field data quality issue is discovered.

Communication Drivers	Responsible Person Affiliation	Name	Phone Number and/or E-Mail	Procedure
Laboratory analytical data quality issues	Laboratory PM Laboratory PM Tetra Tech Project Chemist Tetra Tech PM Navy RPM	Kim Kostzer Lori Palmer Matt Kraus Geoff Pope Sarah Reed	615-345-1115 813-881-9401 412-921-8729 901-523-9500 904-452-6290	The Laboratory PM will notify the Tetra Tech Project Chemist (verbally or via e-mail) within one business day of identification of a problem related to laboratory data. The Tetra Tech Project Chemist will notify the Tetra Tech PM and the data validation staff (verbally or via e-mail) within one business day. The Tetra Tech PM will notify the Navy RPM (verbally or via e-mail) of significant data quality issues within one business day of resolution.

SAP Worksheet #7 -- Personnel Responsibilities and Qualifications Table
(UFP-QAPP Manual Section 2.4.3)

The personnel and the analytical laboratory responsible for implementing the SAP are identified in the following table. Resumes are available upon request.

Name	Title/Role	Organizational Affiliation	Responsibilities
Sarah Reed	RPM/ Manages project activities for the Navy	NAVFAC Southeast	Oversees project implementation, including scoping, data review, and evaluation.
Greg Campbell	IRP Manager/ Saufley Field POC/ Manages daily site activities related to this project	NAS Pensacola	Oversees site activities and participates in scoping, data review, evaluation, and reviews the SAP.
David Grabka	PM/ Provides regulatory input	FDEP	Participates in scoping, data review, evaluation, and approves the SAP on behalf of FDEP.
Geoff Pope	PM/ Manages project on a daily basis	Tetra Tech	Oversees project, financial, schedule, and technical day-to-day management of the project.
Yarissa Martinez	FOL/SSO Manages field operations and site safety issues	Tetra Tech	Supervises, coordinates, and performs field sampling activities. As SSO, is responsible for training and monitoring site conditions. Details of these responsibilities are presented in the site-specific HASP.
Tom Johnston	QAM/ Oversees program and project QA activities	Tetra Tech	Reviews the SAP and ensures quality aspects of the CLEAN program are implemented, documented, and maintained,
Matt Soltis	HSM/ Oversees health and safety activities	Tetra Tech	Oversees Tetra Tech CLEAN Program Health and Safety Program.

Name	Title/Role	Organizational Affiliation	Responsibilities
Matt Kraus	Project Chemist/ Conducts data validation and reporting	Tetra Tech	Participates in project scoping, prepares laboratory scopes of work, and coordinates laboratory-related functions with laboratory. Oversees data quality reviews and QA of data validation deliverables.
Joseph Samchuck	DVM/ Oversees data validation activities	Tetra Tech	Manages data validation activities within Tetra Tech, including ensuring QA of data validation deliverables, providing technical advice on data usability, and coordinating and maintaining the data validation review schedule.
Lee Leck	Data Manager/ Manages databases	Tetra Tech	Manages Tetra Tech databases and ensures correct input of data.
Kim Kostzer Lori Palmer	Laboratory PM/ Representative for Laboratory and Analytical Issues	Empirical SunLabs	Coordinates analyses with laboratory chemists, ensures that scope of work is followed, provides QA of data packages, and communicates with Tetra Tech project staff.
TBD	DPT Driller Subcontractor PM/ Provides DPT Services	TBD	Ensures that project specific requirements are communicated to field personnel.
TBD	Well Installation Subcontractor PM/ Provides Well Installation Services	TBD	Ensures that project specific requirements are communicated to field personnel.

In some cases, one person may be designated responsibilities for more than one position. For example, the FOL may be responsible for SSO duties. This action will be performed only as credentials, experience, and availability permits.

SAP Worksheet #8 -- Special Personnel Training Requirements Table
(UFP-QAPP Manual Section 2.4.4)

Each site worker will be required to have completed a 40-hour course (and 8-hour refresher, if applicable) in Health and Safety Training as described under Occupational Safety and Health Administration (OSHA) 29 Code of Federal Regulations (CFR) 1910.120(b)(4). Safety requirements will be addressed in greater detail in the site-specific HASP.

SAP Worksheet #9 -- Project Scoping Session Participants Sheet
(UFP-QAPP Manual Section 2.5.1)

Project Name: Saufley Field, Site 5 Projected Date(s) of Sampling: October 2010 through February 2011 Project Manager: Geoff Pope [1]	Site Name: Site 5 Site Location: Saufley Field, Pensacola, FL

Date of Session: April 12, 2010
Scoping Session Purpose: Pre-DQO meeting to discuss current data gaps.

Name	Title	Affiliation	Phone #	E-Mail Address	Project Role
William Gates	RPM	NAVFAC Southeast	843-763-5177	w_gates@bellsouth.net	Manages Project Activities for the Navy
Greg Campbell	Saufley Field POC	NAS Pensacola	850-452-3131	gregory.campbell@navy.mil	Environmental Coordinator
Gerry Walker	Base Activity Coordinator	Tetra Tech	850-385-1362	gerry.walker@tetratech.com	Manages Base Activities for Tetra Tech
William Wright [1]	PM	Tetra Tech	412-921-8889	william.wright@tetratech.com	Manages Project Activities
Chuck Metz	Project Engineer	Tetra Tech	412-921-8214	charles.metz@tetratech.com	Technical Advisor
Gary Steigel	Project Engineer	Tetra Tech	412-921-8825	gary.stiegel@tetratech.com	Assists in Managing Project Activities
Peggy Churchill	DQO Facilitator	Tetra Tech	321-636-1300	peggy.churchill@tetratech.com	Leads Development of DQOs

Notes: 1 - Geoff Pope replaced William Wright as the Tetra Tech PM for this project.

Comments/Decisions: It was discussed that there was no documented closure of the fuel pipelines at Saufley. Greg remembers closure work occurring by E.C. Jordan about 20 years ago. Site 4 and Site 2406 data should be reviewed so there is no overlap between this investigation and what was conducted at those sites. The question left to be answered was that a site assessment is needed until closure activities occur that result in the need for additional assessment. If the pipeline was never properly closed, closure activities would need to be conducted prior to or concurrently with the site assessment.

Action Items:

1) Chuck Metz to call David Grabka to ask about Saufley documents in FDEP's records.

2) William Wright to check with his validation group to determine data validation requirements (full, limited, or cursory) for petroleum sites.

3) Greg Campbell to look for closure report for Chevalier Field because it included bowsers similar to this site.

4) Chuck to e-mail Greg the NAVFAC drawing number for Saufley plans that contain pipelines.

5) Greg Campbell to talk with Saufley Air Operations to see if they can provide any information on closure dates.

6) Bill Gates will try to find the E.C. Jordan report for the removal and closure of bowers and associated pipelines.

Consensus Decisions: It was agreed that those with action items would complete and report back to the team in approximately two weeks to determine the path forward.

Project Name: Site 5/Saufley Field Projected Date(s) of Sampling: September 13, 2010 through February 25, 2011 Project Manager: Geoff Pope [1]	Site Name: Site 5 Site Location: Saufley Field, Pensacola, FL

Date of Session: April 27, 2010
Scoping Session Purpose: Partnering meeting for Saufley (Discussed historical findings and path forward for investigating the site).

Name	Title	Affiliation	Phone #	E-Mail Address	Project Role
William Gates	RPM	NAVFAC Southeast	843-763-5177	w_gates@bellsouth.net	Manages Project Activities for the Navy
Greg Campbell	Saufley Field POC	NAS Pensacola	850-452-3131	gregory.campbell@navy.mil	Environmental Coordinator
David Grabka	PM	FDEP	850-245-8997	david.grabka@dep.state.fl.us	Provides Regulator Input
Gerry Walker	Base Activity Coordinator	Tetra Tech	850-385-9899 Ext. 1362	gerry.walker@tetratech.com	Manages Base Activities for Tetra Tech
William Wright [1]	PM	Tetra Tech	412-921-8889	william.wright@tetratech.com	Manages Project Activities

Notes: 1 - Geoff Pope replaced William Wright who was initially the Tetra Tech PM for this project.

Comments/Decisions: It was discussed that the pipeline and bowser closure work by E.C. Jordan had been performed, but no closure reports were on file with FDEP or the Navy. Therefore, closure of the pipeline was required and that the Site 5 SAP Tetra Tech was preparing should include this. Documentation of the closure work would be included as an appendix to the Site Assessment Report (SAR).

Action Items: Tetra Tech would proceed with the SAP. An Alternate Sampling Plan (ASP) would be drafted and submitted to FDEP for their review and approval. The sampling requirements of the ASP (provided in Appendix A) would be incorporated into the SAP.

Consensus Decisions: None

Project Name: Saufley Field, Site 5 Projected Date(s) of Sampling: September 13, 2010 through February 25, 2011 Project Manager: Geoff Pope [1]	Site Name: Site 5 Site Location: Saufley Field, Pensacola, FL

Date of Session: June 2, 2010
Scoping Session Purpose: DQO Meeting

Name	Title	Affiliation	Phone #	E-Mail Address	Project Role
William Gates	Navy RPM	NAVFAC Southeast	843-763-5177	w_gates@bellsouth.net	Manages Project Activities for the Navy
Greg Campbell	Saufley Field POC	NAS Pensacola	850-452-3131	gregory.campbell@navy.mil	Environmental Coordinator
David Grabka	PM	FDEP	850-245-8997	david.grabka@dep.state.fl.us	Provides Regulator Input
Gerry Walker	Base Activity Coordinator	Tetra Tech	850-385-9899 Ext. 1362	gerry.walker@tetratech.com	Manages Base Activities for Tetra Tech
William Wright [1]	PM	Tetra Tech	412-921-8889	william.wright@tetratech.com	Manages Project Activities
Gary Stiegel	Assistant PM	Tetra Tech	412-921-8825	gary.stiegel@tetratech.com	Assists in Managing Project Activities
Charles Metz	Project Engineer	Tetra Tech	412-921-8214	charles.metz@tetratech.com	UFP-SAP Development
Peggy Churchill	DQO Facilitator	Tetra Tech	321-636-6470 Ext. 1300	peggy.churchill@tetratech.com	Leads Development of DQOs

Notes: 1 - Geoff Pope replaced William Wright who was initially the Tetra Tech PM for this project.

Comments/Decisions: DQOs were presented to the Project Team in a teleconference. DQOs were accepted as presented in Worksheet #11. There were no major changes that came up, except that the field sampling design would be based on ASP requirements of FDEP's pipeline closure rules (Chapter 62-761 F.A.C.). A couple of minor changes were to perform cursory validation on all data and to include Total Recoverable Petroleum Hydrocarbon (TRPH) speciation via the Total Petroleum Hydrocarbon Criteria Working Group (TPHCWG) method.

Action Items: Procure laboratory and complete SAP.

Consensus Decisions: Decision was made to proceed with an ASP that differs from FDEP sampling requirements for pipeline closure. Details of this plan are provided in Worksheet #11.

SAP Worksheet #10 -- Conceptual Site Model
(UFP-QAPP Manual Section 2.5.2)

10.1 FACILITY BACKGROUND

The general location of Saufley Field is shown on Figure 10-1. Saufley Field is located on the Florida panhandle approximately five miles northwest of Pensacola, Florida. The installation currently encompasses approximately 866 acres and includes four airstrips, of which two are active, and a number of small buildings which are located south of the airfield. A Federal Prison is located south of the airfield adjacent to the Site. The majority of Saufley Field is covered by paved runway surrounded by mowed open grassy fields and infrastructure for tenant support. Approximately 200 of the 866 acres are undeveloped. South of the airstrips, the majority of the adjacent area is predominantly wooded and supports a wide variety of flora and fauna.

Saufley Field is an active military facility that was originally built and subsequently developed further to support various military activities including pilot training and is now used primarily to train and educate Navy personnel and to house federal prisoners. NAS Whiting Field pilots use two of the airstrips for touch and go landing exercises.

Saufley Field was opened in 1940 as a Naval Auxiliary Air Station (NAAS) and was re-designated a NAS in 1968. It was decommissioned in 1976 and designated as an OLF and reactivated in 1979 as a Naval Education and Training Program Development Center and as an OLF for NAS Whiting Field pilot training. In 1996, Saufley Field became the Naval Education and Training Professional Development and Technology Center (NETPDTC), a major shore command. As the host of Saufley Field, NETPDTC supports 10 major Department of Defense (DoD) as well as Navy tenants and has a total base population in excess of 1,000. Saufley Field operates two active runways and has in excess of 34,425 square feet of hangar space.

In 2008, the Navy entered into negotiations to form an Enhanced Use Lease partnership with private industry. The objective of the Enhanced Use Lease program is to transform 104 acres of the property at Saufley Field into a diversified, multi-use business campus through the creative adaptation and reuse of two sites (areas of the base). The first area contained 85.5 acres with 60 buildings (including 4 hangars) encompassing 622,000 square feet of space and the second area contained 18.7 acres that is currently used as a golf course. The total area also offers potential access to two 4,000 linear foot runways.

10.2 PHYSICAL SITE DESCRIPTION

Site 5 consists of the aviation gasoline (AVGAS) and jet fuel (JP-4) fuel distribution lines connecting a UST farm to the flight line and refueling pits, or bowsers, located along the aircraft parking ramp (tarmac)

(Figure 10-2). The UST farm was removed and an environmental investigation was conducted. Site 5 is located in the northwest portion of Saufley Field.

10.3 PREVIOUS ENVIRONMENTAL INVESTIGATIONS AND SITE HISTORY

In late 1950, lightning ignited a row of refueling pits north of Hangars 807 and 808 causing minor damage to the pits involved. It is not known whether any damage was incurred by the fuel lines.

In May 1992, Naval Energy and Environmental Support Activity (NEESA) submitted the "Preliminary Assessment Report, Naval Educational and Training Program Management Support Activity (NETPMSA), Saufley Field, Escambia County, Florida" (NEESA, 1992). In this document, NEESA made the following general statements or observations regarding the entire site that are applicable to Site 5:

- Between 1942 and 1976, numerous types of solvents, oils, and fuels were used at Saufley Field to support air operations.
- By volume, more high octane AVGAS was used than any other hazardous material.
- Exact usage rates of fuels, oils, and solvents at Saufley Field are unknown.
- During aerial operations between 1942 and 1977, 14 USTs and 2 aboveground storage tanks (ASTs) were in service.
- Most of the tanks were removed in the late 1980s.

Specific references to Site 5 are:

- The seven aerial (read as AVGAS) refueling tanks (subject of UST Site 2406 investigation) were connected by over two miles of 10-inch and 8-inch diameter steel fuel lines to 55 refueling pits (bowsers) located on the aircraft parking ramp (tarmac).

Design drawings prepared in the 1940s detail the gasoline distribution system at Site 5 and include details of the piping, bowsers, and the fuel tank farm. From these drawings, the locations of valve pits, joints, tees, bleeders, and an expansion loop could be identified. Drawings prepared in 1985 (NAVFAC DWG #s 5134920 thru 5134925) for the pipeline capping and tank closure at Saufley Field were also located. The design drawings were prepared by the Navy PWC design section and show following:

- The six 25,000-gallon fuel tanks (AVGAS) and the one 15,000 gallon fuel tank (JP-4) were removed and the existing fuel lines were capped and abandoned-in-place at the fuel tank farm.
- The 500-gallon lube oil tanks located under bowsers were removed.
- The pipes were capped at the fuel pipeline valve pits.
- Trenches and service pits were filled and paved with concrete.
- As-built revision dated June 1, 1987 indicates that the contractor performed work in accordance with the design drawings.

E.C. Jordan is believed to have performed the closure action on the bowsers. However, no closure documentation has been located. A review of the closure design drawings in April 2010 revealed that the fuel lines and bowsers located within the concrete tarmac area had once been located in concrete trenches and pits covered with steel plating. Per the closure design drawings, all bowser pits and fuel line trenches were filled with compacted soil and covered with 6-inches of concrete to grade level.

No closure documentation could be located in the facility environmental files. No environmental investigations involving sampling and analysis of environmental media have been performed at Site 5.

10.4 CONCEPTUAL SITE MODEL

A conceptual site model (CSM) that provides a plan view of the source area, stratigraphy, hydrogeology, and contamination migration pathways is provided on Figure 10-3. The environmental media potentially affected by releases to the environment include:

- Subsurface soil – Contaminants released to the subsurface soils may exceed their properties for leachability as a result of fuel spills.
- Groundwater – Contaminants released to the subsurface soils may exceed their properties for leachability and migrate to groundwater as a result of fuel spills.

10.4.1 Geology and Hydrogeology

The soil in the area of Site 5 consists of Lakeland sand which is very permeable; therefore, AVGAS and JP-4 may have leached into the soil. The aircraft parking ramp (tarmac) is flat, but the surrounding area drains to the northwest. Surface water runoff from the general site area drains toward Eight Mile Creek (a branch of Eleven Mile Creek), which is located to the north-northwest of Site 5.

In the southern half of Escambia County, the sand and gravel aquifer and the upper limestone of the Floridan aquifer are separated by a thick section of relatively impermeable clay; but in the northern half, the sand and gravel aquifer and the upper limestone of the Floridan aquifer are in contact with one another. The upper limestone of the Floridan aquifer is separated from the lower limestone by a thick clay bed (Musgrove et. al., 1965).

The sand and gravel aquifer is composed of sand with numerous lenses and layers of clay and gravel. The formation also contains lenses of hardpan where the sand has been cemented by iron oxide minerals. This aquifer lies at the surface throughout Escambia County. Logs of borings from various locations throughout Saufley Field show that the surficial sands extend from ground surface to a depth of at least 129 feet above mean sea level (msl), below which is a 15-feet thick marine clay, the continuity of which is uncertain. Underlying the clay is more sand with numerous clay lenses (Geraghty and Miller, 1986).

Water levels in the shallow aquifer range from 27 feet (near the southeastern perimeter of the facility) to approximately 50 feet below ground surface (bgs) near the western edge of the facility. The groundwater flow has historically been toward the Gulf of Mexico and Escambia and Perdido Rivers; however, groundwater flow can vary locally due to the effect of topography or surface water bodies. Also, the aquifer recharge is predominantly from local precipitation (Trapp, 1973).

The shallow saturated permeable beds in the sand and gravel aquifer contain groundwater under non-artesian conditions, while the deeper permeable beds contain groundwater under artesian pressure, where they are confined by lenses of clay and sandy clay (NEESA, 1983).

Below the sand and gravel aquifer, the limestone layers comprise the regionally extensive Floridan aquifer, which in this area is divided into upper and lower units separated by the Bucatunna clay. The upper Floridan aquifer is an important source of water in areas east of Escambia County; however, in the Pensacola area it is highly mineralized and not used as a water supply. The lower Floridan aquifer is also highly mineralized and is designated for use as an injection zone (Geraghty and Miller, 1986).

Groundwater flow at Site 5 will be determined as part of the proposed sampling event.

10.4.2 Nature and Extent of Contamination

Currently, assessment activities have not been conducted nor have environmental samples (soil and groundwater) been collected from Site 5. The investigation has two purposes. The initial purpose of the investigation activities is to collect data to determine whether there is any unacceptable environmental contamination at Site 5 associated with the fuel distribution system and to generate a closure report under Chapter 62-761 F.A.C. If no contamination is found, the closure report will recommend no further action (NFA). If contamination is found, the closure report will recommend a site assessment be conducted to delineate contamination and a Site Assessment Report (SAR) be conducted under 62-770 F.A.C. The data collected will be used to evaluate the nature and extent of chemicals detected in soil and groundwater and presented in the SAR. If soil and groundwater do not exceed the FDEP cleanup target levels (CTLs), then a No Further Action (NFA) decision is warranted and no remedial action would be necessary.

10.4.3 Migration Pathways

Contaminants released to the environment from fuel spills or leaks from the fuel distribution system may have adversely affected subsurface soils beneath and adjacent to Site 5 and if they exceed their properties for leachability, they could migrate to groundwater during precipitation events.

10.4.4 Potential Receptors

Human receptors potentially include: industrial workers, construction workers, maintenance workers, trespassers/recreational users, and hypothetical future residents. However, because the current and future industrial use is not anticipated to change, maintenance workers and trespassers are considered to be the most likely receptors to contact contaminants that may be present in subsurface soil at Site 5. The assumed exposure routes for contact with the subsurface soil for the anticipated receptors include: ingestion, dermal contact, and inhalation.

In 1994, the PWC potable water treatment system at Saufley Field included two active potable water (PW) wells. On May 9, 1994, a water sample from PW04 effluent indicated benzene concentrations of 32 micrograms per liter (µg/L), exceeding the FDEP drinking water standard of 1 µg/L. PW04 was taken off-line and was subsequently placed on quarterly sampling for one year for observation and corrective action to remove the contamination. In April 1996, potable water wells PW03 and PW04 were abandoned in-place. Currently, the only source of potable water for Saufley Field is a well field located at the Naval Technical Training Center (NTTC) Corry Station, located approximately 5.5 miles south of the installation. Therefore, groundwater from the Site is not used as a water supply; however, an assumed exposure route to the hypothetical future resident for contact with groundwater exists.

Saufley Field is surrounded by a perimeter security fence; however, a separate fence or other barrier is not provided for Site 5. Access to the installation is restricted to Navy and civilian personnel, authorized contractors, and visitors.

10.4.5 Current and Potential Future Land Uses

Saufley Field is an active military facility that was originally built and subsequently developed further to support various military activities including pilot training and is now used primarily to train and educate Navy personnel and to house federal prisoners. NAS Whiting Field pilots use two of the airstrips for touch and go landing exercises.

Currently, the primary mission of this facility is tenant support, which includes an Enhanced Use Lease partnership with private industry. Additional missions include use as an emergency landing location and land use at Saufley Field is considered to be military/industrial. The site is expected to remain in use for aircraft operations, educational purposes, and as a correctional facility into the future.

There are no known future land use/development restrictions identified for Saufley Field.

SAP Worksheet #11 -- Project Quality Objectives/Systematic Planning Process Statements
(UFP-QAPP Manual Section 2.6.1)

The following text describes the development of the Project Quality Objectives (PQOs) using USEPA's DQO (System Planning) Process.

11.1 PROBLEM DEFINITION

The primary objective of this investigation is to first determine if contaminants are present within the footprint of the pipeline system and former bowser locations, and if contamination exists at levels that exceed risk-based screening levels. The secondary objective is to determine the nature and extent of contamination in accordance with FDEP guidelines. A pipeline closure assessment report will document if contamination is present and, if necessary, a SAR will document the nature and extent of contamination.

Environmental data is required to refine the CSM and prepare a closure report and/or SAR for Site 5. The report will determine if subsurface soil and groundwater within the boundary of Site 5 have been affected by potential contaminants that are related to the fuel distribution system. The environmental data will be used to characterize the nature and extent of contaminants present in subsurface soil and groundwater within the boundary of Site 5.

11.2 INFORMATION INPUTS

This sampling effort will utilize a Triad Approach to collect, evaluate, and prioritize data collection to evaluate the extent of contaminants in subsurface soil and groundwater. A DPT rig in combination with field screening instruments and a mobile lab will be used to collect data for real-time decision making. Field screening analysis of volatile organic vapors by a flame ionization detector (FID) and of Total Petroleum Hydrocarbons (TPH) by an ultraviolet fluorescence (UVF) detector will be used to select samples (subsurface soil and groundwater) for off-site confirmation analysis and to select the locations of monitoring wells that will be used to collect groundwater samples. If necessary, a mobile laboratory will be used to delineate groundwater.

The following physical and chemical data will be collected during this investigation:

1. FID: The FID is a screening tool that is designed for sensitivity to straight chain hydrocarbons. The FID will be used for volatile organic vapors field screening.

2. UVF 3100: The UVF 3100 is a screening tool with semi-quantitative capabilities for TPH and/or polycyclic aromatic hydrocarbons (PAHs). The UVF 3100 is designed for sensitivity to two or three ring aromatic compounds, or PAHs, which fluoresce when exposed to certain wavelengths of light.

3. Chemical Data: Subsurface soil and groundwater samples will be analyzed by Empirical for the select list of target analytes that are presented in Worksheet #15. Additionally select subsurface soil samples will be analyzed by Sunlabs for TPH via the TPHCWG method, Carbon ranges to be quantitated that are presented in Worksheet #15. The project team accepts that the C5 to C7 range actually represents "post n-pentane" through C7 as a result of using pentane as the extraction solvent. The sampling methods that will be utilized are presented in Worksheet #18, and the analytical methods are presented in Worksheet #19.

4. Field Parameters: Field investigation parameters for groundwater will include dissolved oxygen (DO), oxidation-reduction potential (ORP), pH, conductivity, temperature, and turbidity. These data will be collected in the field. The relevant Standard Operating Procedures (SOPs) are presented in Worksheet #21.

5. Groundwater Level Measurements: Synoptic groundwater levels will be measured in each monitoring well to determine the groundwater flow direction. The sampling methods are presented in Worksheet #18.

Project Action Limits (PALs): Concentrations of target analytes will be compared against PALs. The PALs for this investigation are derived from the following criteria for each media of concern:

Soil

- Soil Cleanup Target Levels (SCTLs) for Chapter 62-777, F.A.C., Table II (Soils) – direct exposure and leachability.

- The laboratory Practical Quantitation Limit (PQL) should be used if it is less stringent than the CTL according to Chapter 62-780.680(2)(b)2.a.(III), F.A.C. The PQL, as defined by the FDEP, is the lowest concentration that a laboratory can accurately report on a chemical.

Groundwater

- FDEP Groundwater Cleanup Target Levels (GCTLs) per Chapter 62-777, F.A.C., Table 1 (Groundwater).

- The laboratory PQL should be used if it is less stringent than the CTL according to Chapter 62-780(1)(c), F.A.C.

11.3 STUDY AREA BOUNDARIES

The horizontal boundaries of Site 5 include any area within a 3 feet radius of the pipelines and all bowsers. Subsurface soil from 2 feet bgs to 10 feet below the last positive (greater than 10 parts per million [ppm]) FID detection or the top of the water table (whichever is encountered first) comprise the vertical soil boundaries. The vertical groundwater boundary is 10 feet below the last positive mobile lab detection, starting from the groundwater interface (assumed to be 40 feet bls).

Soil samples will be collected until positive FID results are no longer detected (or until groundwater is intercepted). Groundwater is estimated to be 40 feet bgs. Groundwater samples will be collected from a screened interval that is placed between the water table to five feet below the water table for analysis. If the soil and groundwater screening criteria is exceeded, then the Site 5 boundary will be expanded by stepping out approximately 20 feet for soil and 100 feet for groundwater in each cardinal direction.

11.4 ANALYTIC APPROACH

The goals of the proposed study are to determine whether contaminant concentrations in soil and/or groundwater within footprint of pipelines and bowsers exceed applicable CTLs. If so, the nature and extent of contamination will be delineated during the initial field investigation. If not, NFA is required.

The confirmation subsurface soil samples and groundwater samples will be analyzed for target analytes based on the requirements identified in Table B of Chapter 62-770, F.A.C. by Empirical, a DoD Environmental Laboratory Accreditation Program (ELAP) accredited and Florida Department of Health (FDOH) National Environmental Laboratory Accreditation Program (NELAP)-certified laboratory. The results of the environmental sampling of subsurface soil and groundwater will be conducted to establish the boundaries of Site 5.

11.4.1 <u>Subsurface Soil Decision Rule</u>

Individual subsurface soil concentrations will be compared to soil PALs.

- If all subsurface soil target analyte concentrations do not exceed their PALs, recommend NFA for soils. Target analytes for soil are limited to the volatile organic compounds (VOCs) of benzene, toluene, ethylbenzene, and total xylenes (BTEX) and methyl tert-buyl ether (MTBE); PAHs; and TRPH, as identified in Worksheet #15.

- If any subsurface soil target analyte concentration exceeds the PAL for that target analyte, then collect additional data via step out samples. Step out samples will be collected until the individual surface soil target analyte concentration from the step out sample no longer exceeds the PAL. If three soil step outs are performed and contamination is not delineated, make adjustments to the sampling strategy to optimize the delineation process.

- If any subsurface soil target analyte concentration is less than the PAL for that analyte (or group of analytes) for step out samples, then the soil contamination has been delineated for that analyte (or group of analytes) and the results will be evaluated in the SAR.

11.4.2 Groundwater Decision Rule

- If all groundwater target analyte concentrations do not exceed their PALs for target analytes, then recommend NFA for groundwater. The target analytes for groundwater are VOCs, including BTEX, MTBE, 1,2-dichloroethane, and other Priority Pollutant List (PPL) Volatile Organic Halocarbons (VOHs); 1,2-dibromoethane (a.k.a. ethylene dibromide, EDB); PAHs; total lead; and TRPH, as identified in Worksheet #15.

- If any groundwater target analyte concentration exceeds the PAL for that target analyte, then collect additional data via step out samples until the individual groundwater target analyte concentration from the step out sample no longer exceeds the PAL. If three groundwater step outs are performed and contamination is not delineated, make adjustments to the sampling strategy to optimize the delineation process.

- If any groundwater target analyte concentration is less than the PAL for that analyte (or group of analytes) for step out samples, then the groundwater contamination has been delineated for that analyte (or group of analytes) and the results will be evaluated in the SAR.

11.4.3 Step Out Samples Decision Rule

Soil

- If soil from the step out sample is less than 10 ppm (corrected for methane interference) on the FID, stop delineating.
- If soil from the step out sample exceeds 10 ppm (corrected value) on the FID, step out 20 feet in each cardinal direction for additional samples.

Groundwater

- If groundwater contaminant concentrations from the step out sample are less than PALs, stop delineating.

- If groundwater contaminant concentrations from the step out sample exceed PALs, step out 100 feet in each cardinal direction for additional samples.

11.5 MEASUREMENT AND PERFORMANCE CRITERIA

Simple comparisons of measured concentrations from biased sampling locations to action levels will be used for the first stages of decision making. The Project Team will use the measured results to determine whether the amount and type of data collected are sufficient to support the attainment of the project objectives. This will involve an evaluation of contaminant concentrations and an evaluation of uncertainty for contaminants that have action levels which are less than the laboratory method detection limits (MDLs), Limits of Detection (LODs), and Limits of Quantitation (LOQs) to ensure that contaminants are likely to have been detected, if present. If all data have been collected as planned and no data points are missing or rejected for quality reasons, then the sampling event completeness will be considered satisfactory. If any data gaps are identified, including missing or rejected data, the Project Team will assess whether a claim of having obtained project objectives is reasonable. This assessment will depend on the number and type of identified data gaps; therefore, a more detailed strategy cannot be presented. All Project Team members will be involved in rendering the final conclusion regarding adequacy of the data.

11.6 PLAN FOR OBTAINING DATA

The soil and groundwater sampling design, rationale, and locations are summarized in Worksheets #17 and #18. These worksheets identify the locations that are to be sampled and the analyses to be conducted.

SAP Worksheet #12 -- Measurement Performance Criteria Table – Field Quality Control Samples

(UFP-QAPP Manual Section 2.6.2)

Quality Control (QC) Sample	Analytical Group	Frequency	Data Quality Indicators (DQIs)	Measurement Performance Criteria (MPCs)	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Equipment Rinsate Blanks	All Fractions	One per 20 field samples per matrix per sampling equipment[1].	Accuracy/ Bias/ Contamination	No analytes ≥ ½ LOQ, except common laboratory contaminants, which must be < LOQ.	S & A
Trip Blanks	VOCs	One per cooler containing VOC samples.	Accuracy/ Bias/ Contamination	No analytes ≥ ½ LOQ, except common laboratory contaminants, which must be < LOQ.	S & A
Field Duplicates	All Fractions	One per 10 field samples collected.	Precision	Values > 5X LOQ: Relative Percent Difference (RPD) ≤30%[2, 3].	S & A
Cooler Temperature Indicator	All Fractions	One per cooler.	Representativeness	Temperature must be less than 6 degrees Celsius (<6 °C).	S

Notes:

1 – Equipment rinsate blanks will be collected if non-dedicated sampling equipment is used. For disposable equipment, one sample per batch of disposable equipment will be collected.
2 – If duplicate values for non-metals are < 5x LOQ, the absolute difference should be < 2x LOQ.
3 – If duplicate values for metals are < 5x LOQ, the absolute difference should be < 4x LOQ.

SAP Worksheet #13 -- Secondary Data Criteria and Limitations Table
(UFP-QAPP Manual Section 2.7)

Secondary Data	Data Source (originating organization, report title and date)	Data Generator(s) (originating organization, data types, data generation / collection dates)	How Data Will Be Used	Limitations on Data Use
Preliminary Assessment	Preliminary Assessment Report, NETPMSA, Saufley Field, Escambia County, Florida	NEESA, 1992	Site 5 location is identified.	None
Design Drawings	NAVFAC Drawing #s 5134920 thru 5134925	NAVFAC, 1985	Soil boring locations are based on the design drawings.	None

SAP Worksheet #14 -- Summary of Project Tasks
(UFP-QAPP Manual Section 2.8.1)

The field tasks are summarized below. A short description of these tasks is also provided.

- Mobilization/Demobilization
- Utility Clearance
- DPT Soil and Groundwater Sampling
- Monitoring Well Installation
- Monitoring Well Sampling
- Monitoring Equipment Calibration
- Surveying
- Investigation-Derived Waste (IDW) Management
- Field Decontamination Procedures
- Field Documentation Procedures

Additional project activities include the following tasks:

- Analytical Tasks
- Data Management
- Data Assessment and Oversight
- Data Reviews
- Project Reporting

A summary of each task is discussed in this worksheet.

14.1 MOBILIZATION/DEMOBILIZATION

A field team orientation meeting will be conducted prior to the start of fieldwork to familiarize the team personnel with the site's health and safety requirements, objectives and scope of the field activities, and chain-of-command. This meeting will be attended by the Tetra Tech FOL/SSO, PM, and Project Chemist. Mobilization activities will include transporting field personnel, equipment, and supplies to the site. All drilling equipment and sampling tools will be cleaned prior to arrival on-site. A 1-hour health and safety meeting will be conducted prior to initiating on-site activities. All subcontractor personnel (including substitutes) will attend the meeting. Tetra Tech will coordinate with the Navy POC at Saufley Field regarding security and access issues, and daily activities. Tetra Tech will also coordinate with the Navy RPM and stakeholders regarding the field activities. Demobilization will include transporting personnel, field equipment, and supplies from the site, performing general site cleanup, and organizing and finalizing field paperwork.

14.2 UTILITY CLEARANCE

Prior to the commencement of any intrusive activities, Tetra Tech will coordinate utility clearance with the Facility and with the Florida One Call system representative. The Facility and Utility Companies will identify and mark-out utilities that may be present within the proposed well installation areas. Subsurface utilities will also be cleared by the well installation subcontractor by notifying the One Call utility clearing service. See Tetra Tech SOP HS-1.0 (Appendix B) on conducting well installations for further information.

14.3 CONCRETE CORING

Prior to commencement of drill activities, concrete and asphalt coring will be required for sampling locations located on asphalt or concrete. A subcontractor will be used to core sample locations in tarnac concrete while the drilling subcontractor should be able to core non tarmac concrete and asphalt locations. Core size is anticipated to be 3 inches in diameter but will be dependent upon the drilling subcontractors sample tools. Typical thicknesses for tarmac coring should be assumed to be between 12 and 24 inches. The concrete may be reinforced with re-bar and should be assumed to contain river rock. Asphalt and concrete thickness for non tarmac areas will likely be less than 4 inches. All cores will be left in place until removed for sampling activities. Following completion of sampling at cored locations, all core hole holes will filled to match existing grade (asphalt or concrete).

14.4 DPT BORING/SUBSURFACE SOIL AND GROUNDWATER SAMPLING

Soil borings will be conducted by DPT in accordance with Tetra Tech SOP SA-2.5 (Appendix B) and subsurface soil samples will be sampled and screened continuously at 2-foot intervals from 2 feet bgs to 10 feet bgs and at 5-foot intervals from 10 feet bgs until the top of the water table (approximately 40 feet bgs), or until two consecutive readings from below the landmark being sampled are clean (activated carbon filter corrected FID readings less than 10 ppm in accordance with FDEP procedures). The soil will be described by the Site Geologist and will be screened for evidence of contamination with an FID following Tetra Tech SOP SA-2.4 (Soil Gas Sampling). Any qualitative signs of potential contamination (such as odor or staining) will be noted. Soil drilling procedures are discussed in Tetra Tech SOP SA-1.3, and soil logging procedures are documented in Tetra Tech SOP GH-1.5. These SOPs are included in Appendix B.

Soil samples will be collected with a 5-foot long DPT core barrel lined with acrylic sleeves at depths greater than 2 feet bgs. The sample aliquots will be collected at prescribed intervals. Each sample aliquot will be screened immediately with an FID, and then transferred to laboratory-supplied sample containers. The samples will be labeled, preserved on ice, and transported to the laboratory. All portions of the sampling equipment used in sample collection will be decontaminated before each use using standard decontamination procedures. Equipment rinsate blanks will be collected from the decontaminated sampler at the prescribed frequency. Soil samples will be collected using the procedures specified in FDEP SOP FS-3000 (Appendix B).

Groundwater samples collected by DPT will be retrieved from stainless steel screen point samplers using either a bailer or low-flow purging techniques (typically at a rate of less than 1 liter per minute) with an appropriate pump and tubing. If possible, prior to sample collection, water will be purged from the screen point until the water is sediment-free or until it is determined the turbidity level is at the lowest value attainable. Groundwater samples will be collected using the procedures specified in FDEP SOP FS-2000 and FS-2200 (Appendix B).

14.5 MONITORING WELL INSTALLATION

If groundwater contamination is found to exist, permanent monitoring wells will be required by FDEP. Otherwise, no permanent monitoring wells may be required. The exact number (maximum of 35) and location of monitoring wells to be installed will be determined by the Partnering Team following review of the DPT groundwater data. All permanent monitoring wells will be installed in accordance with the local Florida water management district rules and permits. Permanent monitoring wells will be installed using either DPT, rotosonic, mud-rotary, or hollow stem auger drilling techniques.

Small diameter monitoring wells (microwells) with 3/4-inch to 1-inch inside diameters (ID), constructed with pre-packed well screens, will be installed by DPT methods where possible. If it is necessary to install conventional monitoring wells by rotosonic, mud rotary, or hollow stem auger methods due to subsurface conditions or the mandate for aquifer testing, such wells will be constructed of 2-inch ID Schedule 40, flush joint polyvinyl chloride (PVC) riser and flush-joint factory slotted well screen. Each section of casing and screen shall be National Sanitation Foundation (NSF) approved. Screen slot size shall be 0.010 inch. Shallow monitoring wells will be constructed with 10 to 15 feet of screen with the top of the screen positioned approximately 4 feet above the water table. Boreholes for 2-inch diameter wells will have a minimum diameter of 6 inches.

Clean silica sand of U.S Standard Sieve Size No. 20/30 will be installed into the boring annulus around the well screen. The sand pack will be set from the bottom of the hole to approximately 2 feet above the top of the well screen. A minimum 2-foot thick 30/65 fine sand seal will be installed above the sand pack.

The remainder of the boring will be backfilled with a Type I Portland cement/bentonite grout. The depths of all backfill materials will be constantly monitored during the well installation process by means of a weighted stainless steel or fiberglass tape. The position of the top of the screened interval, sand pack, and fine sand seal may be adjusted as site conditions warrant (elevated water table, etc.).

If any monitoring well boreholes will potentially pass through contaminated zones or confining layers, an outer protective casing will be installed to prevent cross contamination of the aquifer below. The outer casing will penetrate the confining layer 1 or 2 feet or be set below the zone of known contamination. Upon completion of the boring, the casing will be set to the desired depth and the annular space filled with Portland cement grout from bottom to top through a tremie pipe. After allowing the grout to cure for a minimum of 24 hours, the mud-rotary drilling method will be used to drill through the outer casing to advance the boring to the desired depth. Double-cased monitoring well construction details (i.e., screen slot size, filter pack, seal, and grout) will be similar to other wells. Rotosonic drilling intrinsically incorporates an outer casing and would preclude an installed casing if used.

Flush mounted steel well covers and manholes will be installed around the monitoring wells. The manhole will consist of a flush-mounted, 22-gauge steel, water resistant, welded box with 3/8-inch thick steel lid. A 2-feet by 2-feet by 6-inch thick concrete apron will be constructed around the manhole. The manhole shall be completed 1 inch above existing grade in grassy areas and the apron tapered to be flush with the existing grade at the edges enabling the water to run off the apron. The manhole shall be completed at grade in paved areas. A detail of a typical flush-mounted monitoring well is provided in Appendix B. All locks supplied for the wells will be keyed alike. After installation, the ground surface and the top of the PVC riser pipe will be surveyed to within 0.01-foot vertical accuracy using datum points as discussed in Section 14.7. A monitoring well construction diagram will be completed for each well installed. A sample of the monitoring well construction form is provided in Appendix B.

The monitoring wells will be developed no sooner than 24 hours after installation to remove fine material from around the screened interval of the well. Wells will be developed by bailing and surging, or by pumping, as determined by the field geologist. Temperature, pH, specific conductance and turbidity will be recorded periodically during development to document stability.

14.6 MONITORING WELL SAMPLING

All groundwater samples collected from monitoring wells will be collected using the procedures specified in FDEP SOP FS 2200 (Appendix B). If Light Non-Aqueous Phase Liquid (LNAPL) is detected in purge water initially being pumped from a monitoring well at any given location, its presence will be noted, but groundwater samples will not be collected for laboratory analysis.

14.7 MONITORING EQUIPMENT CALIBRATION

Monitoring equipment calibration procedures are described in Worksheet #22.

14.8 SURVEYING

The locations of sample points, soil borings, and monitoring wells may initially be determined during the field investigation using a portable global positioning system (GPS) instrument with sub-meter accuracy. This information may be helpful in plotting results and analyzing the data coverage in real-time to make data acquisition decisions during the investigation. The GPS instrument will be used in accordance with Tetra Tech SOP-05 (Appendix B), and results will be recorded in the field logbook. Monitoring wells and other selected points, however, will be permanently located using a National Geodetic Vertical Datum (NGVD) survey at the conclusion of field activities.

If monitoring wells are installed, the locations of monitoring wells will be measured by a certified land surveyor. Each point will be measured from a reference location tied to the Florida State Plane Coordinate System. An X-Y coordinate system shall be used to identify locations. The X-coordinate will be the east-west axis and the Y-coordinate will be the north-south axis.

All survey locations will be reported using the Florida State Plane Coordinate system. Existing installation benchmarks will serve as horizontal and vertical datums for the survey. Elevations and horizontal locations will be recorded to the nearest hundreds of a foot. The elevations of all monitoring wells will be surveyed at the water level measuring reference point on top of the well riser and on the undisturbed ground surface adjacent to the well pad.

14.9 INVESTIGATION-DERIVED WASTE MANAGEMENT

It is anticipated that IDW materials will be generated during the field investigation, including potentially soil cuttings from the monitoring well development, and aqueous fluids from decontamination, purge, and development water. These wastes must be disposed in such a manner that does not contribute to further environmental contamination or pose a threat to public health or safety. Tetra Tech SOP SA-7.1 (Appendix B) provides information on the handling of IDW. Drums for storage of IDW will be provided by Saufley Field. Disposal of the IDW following receipt of the analytical data should be coordinated with Saufley Field.

14.10 FIELD DECONTAMINATION PROCEDURES

Decontamination of major equipment and sampling equipment will be in general accordance with FDEP SOP FC-1000 (Appendix B). Sampling equipment will be decontaminated prior to and between sampling at each location. At each site, an abbreviated decontamination procedure consisting of a soapy water (laboratory-grade detergent) rinse followed by a deionized (DI) water rinse will be performed. Decontamination fluids (IDW) will be collected in 5-gallon buckets and transferred into drums for disposal following the procedures listed in Section 14.8 (IDW Management).

14.11 FIELD DOCUMENTATION PROCEDURES

Pre-preserved, certified-clean bottle ware will be supplied by the laboratories. Matrix-specific sample log sheets will be maintained for each sample collected. In addition, sample collection information will be recorded in bound field notebooks or specific field forms. Samples will be packaged and shipped according to FDEP SOP FS-1000 (Appendix B).

Field documentation will be performed in accordance with Tetra Tech SOP SA-6.3 (Appendix B). A summary of all field activities will be properly recorded in indelible ink in a bound logbook with consecutively numbered pages that cannot be removed. Logbooks will be assigned to field personnel and will be stored in a secured area when not in use.

At a minimum, the following information will be recorded in the site logbook:

- Name of the person to whom the logbook is assigned.
- Project name.
- Project start date.
- Names and responsibilities of on-site project personnel including subcontractor personnel.
- Arrival/departure of site visitors.
- Arrival/departure of equipment.
- Sampling activities and sample log sheet references.
- Description of subcontractor activities.
- Sample pick-up information including chain-of-custody numbers, air bill numbers, carrier, time, and date.
- Description of borehole or monitoring well installation activities and operations.
- Health and safety issues.
- Description of photographs including date, time, photographer, roll and picture number, location, and compass direction of photograph.

All entries will be written in indelible ink and no erasures will be made. If an incorrect entry is made, striking a single line through the incorrect information will make the correction; the person making the correction will initial and date the change.

14.12 ANALYTICAL TASKS

TRPH speciation will be performed by SunLabs Inc. using the TPHCGW Method. SunLabs is a FDOH NELAP accredited laboratory for TRPH speciation via the TPHCWG Method. The Navy has elected to perform a project specific evaluation of this laboratory and their SOP for TPHCWG. A copy of SunLab's accreditation can be found in Appendix C.

All other chemical analyses will be performed by Empirical. Empirical is a current DoD ELAP and Florida NELAP accredited laboratory for all analytical groups and target analytes that will be analyzed. A copy of Empirical's accreditation can be found in Appendix C. Analyses will be performed in accordance with the analytical methods identified in Worksheet #19. Empirical is expected to meet the PALs to the extent identified in Worksheet #15. Empirical will perform chemical analysis following laboratory-specific SOPs (Worksheets #19 and #23) developed based on the analytical methods listed in Worksheets #19 and #30. Copies of the Laboratory SOPs are included in Appendix C. All results will be reported by the laboratory on a dry-weight basis. Results of percent moisture will be reported in each analytical data package and electronic data files. This information will also be captured in the project database which will eventually be uploaded to Naval Installation Restoration information Solution (NIRIS). Percent moisture information will be presented in the SAR.

The analytical data packages provided by Empirical and SunLabs will be in a contract laboratory program (CLP)-like format and will be fully validatable and contain raw data, summary forms for all sample and laboratory method blank data, and summary forms containing all method specific QC (results, recoveries, relative percent differences, relative standard deviations, and/or percent differences etc.).

14.13 DATA MANAGEMENT

Data management activities are crucial to ensure reliability and maintain organization of the site data. This section describes essential data management activities which include: data handling, data tracking and control, and record keeping.

14.13.1 <u>Data Handling</u>

After the field investigation is complete, field sampling log sheets will be organized by date and media and filed in the project files. The field logbooks for this project will be used only for this site, and will also be categorized and maintained in the project files after the completion of the field program. Project personnel completing concurrent field sampling activities may maintain multiple field logbooks. When possible, logbooks will be segregated by sampling activity. The field logbooks will be titled based on date and activity. The data handling procedures to be followed by the laboratories will meet the requirements of the technical specification. The electronic data results will be automatically downloaded into the Tetra Tech database in accordance with proprietary Tetra Tech processes. Corrections to entries made in field and laboratory logs will be made by striking through the erroneous entry with a single line and entering the correction nearby with the date of correction and initials of the person making the correction.

14.13.2 Data Tracking and Control

The Tetra Tech PM (or designee) is responsible for the overall tracking and control of data generated for the project.

- **Data Tracking –** A "cradle to grave" sample tracking system will be used from the beginning to the end of the investigation to track and control the data generated during the Site 5 investigation. The Tetra Tech FOL will initiate the sample tracking process by ensuring that sample jar labels are complete and adhere to SAP requirements. When field sampling is underway, the Tetra Tech FOL will forward the chain-of-custody forms to the Tetra Tech PM or designee via fax at the end of the day. The Tetra Tech PM or designee will compare the entries on the chain-of-custody forms with the SAP to confirm that the correct information is being collected/requested. This will allow for early detection of errors made in the field. The Tetra Tech Project Chemist (or designee) is responsible for tracking the samples collected and shipped to the subcontract laboratory. Upon receipt of the data packages from the analytical laboratory, the Tetra Tech Project Chemist will oversee the data validation effort, which includes verifying that the data packages are complete and results for all samples have been delivered by the analytical laboratory.

- **Data Storage, Archiving, and Retrieval –** The data packages received from the subcontract laboratory are tracked in the data validation log book. After the data are validated, the data packages are entered into the Tetra Tech CLEAN file system and archived in secure files. The field records including field log books, sample logs, chain-of-custody records, and field calibration logs will be submitted by the Tetra Tech FOL to be entered into the CLEAN file system prior to archiving in secure project files. The project files are subject to audit to verify accuracy and completeness. At the completion of the Navy contract, the records will be stored by Tetra Tech and eventually handed over to NAVFAC.

- **Data Security –** The Tetra Tech project files are restricted to designated personnel only. Records can only be borrowed temporarily from the project file using a sign-out system. The Tetra Tech Data Manager maintains the electronic data files. Access to the data files is restricted to qualified personnel only. File and data backup procedures are routinely performed.

14.13.3 Record Keeping

A number of documents must be completed before, during, and after the sampling event. These documents include at a minimum: chain-of-custody sheets, field data sheets, field books, field notes, photographs, and analytical data. In addition, adherence to sample holding times, sample preservation, and container requirements must also be documented. Field and analytical documentation will be maintained in the Tetra Tech project database and project file, as presented in Worksheet #29.

14.14 DATA ASSESSMENT AND OVERSIGHT

Refer to Worksheet #32 for assessment findings and corrective actions and to Worksheet #33 for QA management reports.

14.15 DATA REVIEWS

Data verification is described in Worksheet #34. Data validation is described in Worksheets #35 and #36. Usability assessment is described in Worksheet #37.

14.16 PROJECT REPORTING

A Site 5 SAR will be prepared documenting the sampling activities and results of the investigation. The presentation will evaluate the nature and extent of contamination and recommend the path forward for the site.

SAP Worksheet #15 -- Reference Limits and Evaluation Table
(UFP-QAPP Manual Section 2.8.1)

15.1 MATRIX: GROUNDWATER

15.1.1 Analytical Group: Total Lead

Analyte	CAS Number	Project Action Limit (1) (ug/L)	Project Action Limit Reference	Project Quantitation Limit Goal (ug/L)	Empirical		
					LOQ (ug/L)	LOD (ug/L)	MDL (ug/L)
LEAD	7439-92-1	15	FDEP GCTL	5.0	3	3	1.5

15.1.2 Analytical Group: PAHs

Analyte	CAS Number	Project Action Limit (1) (ug/L)	Project Action Limit Reference	Project Quantitation Limit Goal (ug/L)	Empirical		
					LOQ (ug/L)	LOD (ug/L)	MDL (ug/L)
1-METHYLNAPHTHALENE	90-12-0	28	FDEP GCTL	9.3	0.2	0.1	0.05
2-METHYLNAPHTHALENE	91-57-6	28	FDEP GCTL	9.3	0.2	0.1	0.05
ACENAPHTHENE	83-32-9	20	FDEP GCTL	6.7	0.2	0.1	0.05
ACENAPHTHYLENE	208-96-8	210	FDEP GCTL	70	0.2	0.1	0.05
ANTHRACENE	120-12-7	2,100	FDEP GCTL	700	0.2	0.1	0.05
BENZO(A)ANTHRACENE	**56-55-3**	**0.05**	FDEP GCTL	**0.017**	**0.2**	**0.1**	**0.05**
BENZO(A)PYRENE	50-32-8	0.2	FDEP GCTL	0.067	0.2	0.1	0.05
BENZO(B)FLUORANTHENE	**205-99-2**	**0.05**	**FDEP GCTL**	**0.017**	**0.2**	**0.1**	**0.05**
BENZO(G,H,I)PERYLENE	191-24-2	210	FDEP GCTL	70	0.2	0.1	0.05
BENZO(K)FLUORANTHENE	207-08-9	0.5	FDEP GCTL	0.17	0.2	0.1	0.05
CHRYSENE	218-01-9	4.8	FDEP GCTL	1.6	0.2	0.1	0.05
DIBENZO(A,H)ANTHRACENE	53-70-3	0.2	FDEP GCTL	0.067	0.2	0.1	0.05
FLUORANTHENE	206-44-0	280	FDEP GCTL	93	0.2	0.1	0.05
FLUORENE	86-73-7	280	FDEP GCTL	93	0.2	0.1	0.05
INDENO(1,2,3-CD)PYRENE	**193-39-5**	**0.05**	**FDEP GCTL**	**0.017**	**0.2**	**0.1**	**0.05**

Analyte	CAS Number	Project Action Limit [(1)] (ug/L)	Project Action Limit Reference	Project Quantitation Limit Goal (ug/L)	Empirical LOQ (ug/L)	Empirical LOD (ug/L)	Empirical MDL (ug/L)
NAPHTHALENE	91-20-3	14	FDEP GCTL	4.7	0.2	0.1	0.05
PHENANTHRENE	85-01-8	210	FDEP GCTL	70	0.2	0.1	0.05
PYRENE	129-00-0	210	FDEP GCTL	70	0.2	0.1	0.05

15.1.3 Analytical Group: VOCs

Analyte	CAS Number	Project Action Limit [(1)] (ug/L)	Project Action Limit Reference	Project Quantitation Limit Goal (ug/L)	Empirical LOQ (ug/L)	Empirical LOD (ug/L)	Empirical MDL (ug/L)
1,1,1-TRICHLOROETHANE	71-55-6	200	FDEP GCTL	67	1	0.5	0.25
1,1,2,2-TETRACHLOROETHANE	**79-34-5**	**0.2**	**FDEP GCTL**	**0.067**	**1**	**0.5**	**0.25**
1,1,2-TRICHLOROETHANE	79-00-5	5	FDEP GCTL	1.7	1	0.5	0.25
1,1-DICHLOROETHANE	75-34-3	70	FDEP GCTL	23	1	0.5	0.25
1,1-DICHLOROETHENE	75-35-4	7	FDEP GCTL	2.3	1	0.5	0.25
1,2,4-TRICHLOROBENZENE	120-82-1	70	FDEP GCTL	23	1	0.5	0.25
1,2-DICHLOROETHANE	107-06-2	3	FDEP GCTL	1.0	1	0.5	0.25
1,2-DICHLOROPROPANE	78-87-5	5	FDEP GCTL	1.7	1	0.5	0.25
BENZENE	71-43-2	1	FDEP GCTL	0.33	1	0.5	0.25
BROMODICHLOROMETHANE	**75-27-4**	**0.6**	**FDEP GCTL**	**0.20**	**1**	**0.5**	**0.25**
BROMOFORM	75-25-2	4.4	FDEP GCTL	1.5	1	0.5	0.25
BROMOMETHANE	74-83-9	9.8	FDEP GCTL	3.2	1	0.5	0.25
CARBON TETRACHLORIDE	56-23-5	3	FDEP GCTL	1.0	1	0.5	0.25
CHLOROBENZENE	108-90-7	100	FDEP GCTL	33	1	0.5	0.25
CHLORODIBROMOMETHANE	**124-48-1**	**0.4**	**FDEP GCTL**	**0.13**	**1**	**0.5**	**0.25**
CHLOROETHANE	75-00-3	12	FDEP GCTL	4.0	1	0.5	0.25
CHLOROFORM	67-66-3	70	FDEP GCTL	23	1	0.5	0.25
CHLOROMETHANE	74-87-3	2.7	FDEP GCTL	0.90	1	0.5	0.25
CIS-1,2-DICHLOROETHENE	156-59-2	70	FDEP GCTL	23	1	0.5	0.25
CIS-1,3-DICHLOROPROPENE	10061-01-5	NA	None	NA	1	0.5	0.25
ETHYLBENZENE	100-41-4	30	FDEP GCTL	10	1	0.5	0.25

Analyte	CAS Number	Project Action Limit [1] (ug/L)	Project Action Limit Reference	Project Quantitation Limit Goal (ug/L)	Empirical		
					LOQ (ug/L)	LOD (ug/L)	MDL (ug/L)
2-CHLOROETHYL VINYL ETHER	110-75-8	NA	None	NA	5	2.5	1.25
TOTAL XYLENES	1330-20-7	20	FDEP GCTL	6.7	3	1.5	0.75
METHYL TERT-BUTYL ETHER	1634-04-4	20	FDEP GCTL	6.7	1	0.5	0.25
TETRACHLOROETHENE	127-18-4	3	FDEP GCTL	1.0	1	0.5	0.25
TOLUENE	108-88-3	40	FDEP GCTL	13	1	0.5	0.25
TRANS-1,2-DICHLOROETHENE	156-60-5	100	FDEP GCTL	33	1	0.5	0.25
TRANS-1,3-DICHLOROPROPENE	10061-02-6	NA	None	NA	1	0.5	0.25
TRICHLOROETHENE	79-01-6	3	FDEP GCTL	1.0	1	0.5	0.25
TRICHLOROFLUOROMETHANE	75-69-4	2,100	FDEP GCTL	700	1	0.5	0.25
VINYL CHLORIDE	75-01-4	1	FDEP GCTL	0.33	1	0.5	0.25
ACROLEIN	**107-02-8**	**3.5**	**FDEP GCTL**	**1.2**	**5**	**2.5**	**1.2**
ACRYLONITRILE	**107-13-1**	**0.06**	**FDEP GCTL**	**0.020**	**10**	**5**	**2.5**
1,3-DICHLOROPROPENE	**542-75-6**	**0.4**	**FDEP GCTL**	**0.13**	**1**	**0.5**	**0.25**

15.1.4 Analytical Group: EDB

Analyte	CAS Number	Project Action Limit [1] (ug/L)	Project Action Limit Reference	Project Quantitation Limit Goal (ug/L)	Empirical		
					LOQ (ug/L)	LOD (ug/L)	MDL (ug/L)
1,2-DIBROMOETHANE (EDB) [2]	**106-93-4**	**0.02**	**FDEP GCTL**	**0.0067**	**0.03**	**0.02**	**0.01**

15.1.5 Analytical Group: TRPH

Analyte	CAS Number	Project Action Limit [1] (ug/L)	Project Action Limit Reference	Project Quantitation Limit Goal (ug/L)	Empirical		
					LOQ (ug/L)	LOD (ug/L)	MDL (ug/L)
TRPH	NA	5,000	FDEP GCTL	1,700	340	170	85

Notes:

CAS = Chemical Abstracts Service
(1) The PAL is the Chapter 62-777 F.A.C. GCTL for groundwater samples (FDEP, 2005).
(2) EDB will be analyzed by SW-846 Method 8011 to attain lower detection limits in accordance with FDEP requirements.

Bolded rows indicate that the PAL is between the laboratory LOQ and LOD. The Project Team has agreed to accept this data for decision making if results below the LOQ are "J" qualified.

Shaded and Bold row indicate the PAL is less than the LOD; therefore, the Project Team has agreed to replace the PALs with the laboratory LOQs for decision making purposes, as suggested in "Guidance for the Selection of Analytical Methods for the Evaluation of Practical Quantitation Limits" (FDEP, October 2004).

Note that data will be reported at the LOQ and MDL, with non-detected data being the MDL followed by a "U" qualifier as per Florida state regulations. The LOD is presented for completeness and compliance with the Department of Defense Quality Systems Manual for Environmental Laboratories (DoD QSM), Version 4.1 (DoD, 2009).

15.2 MATRIX: SOIL

15.2.1 Analytical Group: PAHs

Analyte	CAS Number	Project Action Limit (1) (mg/kg)	Project Action Limit Reference	Project Quantitation Limit Goal (mg/kg)	Empirical		
					LOQ (mg/kg)	LOD (mg/kg)	MDL (mg/kg)
1-METHYLNAPHTHALENE	90-12-0	3.1	Leach to GW - SCTL	1.0	0.007	0.0033	0.0017
2-METHYLNAPHTHALENE	91-57-6	8.5	Leach to GW - SCTL	2.8	0.007	0.0033	0.0017
ACENAPHTHENE	83-32-9	2.1	Leach to GW - SCTL	0.70	0.007	0.0033	0.0017
ACENAPHTHYLENE	208-96-8	27	Leach to GW - SCTL	9.0	0.007	0.0033	0.0017
ANTHRACENE	120-12-7	2,500	Leach to GW - SCTL	830	0.007	0.0033	0.0017
BENZO(A)ANTHRACENE	56-55-3	0.8	Leach to GW - SCTL	0.27	0.007	0.0033	0.0017
BENZO(A)PYRENE	50-32-8	0.1	Residential - SCTL	0.033	0.007	0.0033	0.0017
BENZO(B)FLUORANTHENE	205-99-2	2.4	Leach to GW - SCTL	0.80	0.007	0.0033	0.0017
BENZO(G,H,I)PERYLENE	191-24-2	2,500	Residential - SCTL	830	0.007	0.0033	0.0017
BENZO(K)FLUORANTHENE	207-08-9	24	Leach to GW - SCTL	8.0	0.007	0.0033	0.0017
CHRYSENE	218-01-9	77	Leach to GW - SCTL	26	0.007	0.0033	0.0017
DIBENZO(A,H)ANTHRACENE	53-70-3	0.1	Residential - SCTL	0.23	0.007	0.0033	0.0017
FLUORANTHENE	206-44-0	1,200	Leach to GW - SCTL	400	0.007	0.0033	0.0017
FLUORENE	86-73-7	160	Leach to GW - SCTL	53	0.007	0.0033	0.0017
INDENO(1,2,3-CD)PYRENE	193-39-5	6.6	Leach to GW - SCTL	2.2	0.007	0.0033	0.0017
NAPHTHALENE	91-20-3	1.2	Leach to GW - SCTL	0.40	0.007	0.0033	0.0017
PHENANTHRENE	85-01-8	250	Leach to GW - SCTL	83	0.007	0.0033	0.0017
PYRENE	129-00-0	880	Leach to GW - SCTL	290	0.007	0.0033	0.0017

15.2.2 Analytical Group: VOCs (BTEX and MTBE Only)

Analyte	CAS Number	Project Action Limit (1) (mg/kg)	Project Action Limit Reference	Project Quantitation Limit Goal (mg/kg)	Empirical		
					LOQ (mg/kg)	LOD (mg/kg)	MDL (mg/kg)
BENZENE	71-43-2	0.007	Leach to GW - SCTL	0.0023	0.005	0.0025	0.00125
ETHYLBENZENE	100-41-4	0.6	Leach to GW - SCTL	0.20	0.005	0.0025	0.00125
METHYL TERT-BUTYL ETHER	1634-04-4	0.09	Leach to GW - SCTL	0.030	0.005	0.0025	0.00125
TOLUENE	108-88-3	0.5	Leach to GW - SCTL	0.17	0.005	0.0025	0.00125
TOTAL XYLENES	1330-20-7	0.2	Leach to GW - SCTL	0.067	0.005	0.0025	0.00125

15.2.3 Analytical Group: TRPH

Analyte	CAS Number	Project Action Limit (1) (mg/kg)	Project Action Limit Reference	Project Quantitation Limit Goal (mg/kg)	Empirical		
					LOQ (mg/kg)	LOD (mg/kg)	MDL (mg/kg)
TRPH	NA	340	Residential - SCTL	110	23	11	6

15.2.4 Analytical Group: TRPH Speciation

TRPH Fraction	CAS Number	Project Action Limit (1) (mg/kg)	Project Action Limit Reference	Project Quantitation Limit Goal (mg/kg)	SunLabs		
					LOQ (mg/kg)	LOD (mg/kg)	MDL (mg/kg)
C5-C7 AROMATICS(2)	**NA**	**34**	**Lowest Calculated SCTL**	**11**	**108**	**TBD**	**27**
>C7-C8 AROMATICS	**NA**	**59**	**Lowest Calculated SCTL**	**20**	**108**	**TBD**	**27**
>C8-C10 AROMATICS	NA	340	Lowest Calculated SCTL	110	108	TBD	27
>C10-C12 AROMATICS	NA	520	Lowest Calculated SCTL	170	108	TBD	27
>C12-C16 AROMATICS	NA	1,000	Lowest Calculated SCTL	330	108	TBD	27
>C16-C21 AROMATICS	NA	1,300	Lowest Calculated SCTL	430	108	TBD	27
>C21-C25 AROMATICS	NA	2,300	Lowest Calculated SCTL	770	108	TBD	27
C5-C6 ALIPHATICS	NA	470	Lowest Calculated SCTL	160	136	TBD	34
>C6-C8 ALIPHATICS	NA	1,300	Lowest Calculated SCTL	430	136	TBD	34
>C8-C10 ALIPHATICS	NA	850	Lowest Calculated SCTL	280	136	TBD	34
>C10-C12 ALIPHATICS	NA	1,700	Lowest Calculated SCTL	570	136	TBD	34
>C12-C8 16LIPHATICS	NA	2,900	Lowest Calculated SCTL	970	136	TBD	34
>C16-C35 ALIPHATICS	NA	42,000	Lowest Calculated SCTL	14,000	136	TBD	34

Notes:

mg/kg = milligrams per kilogram

(1) The PAL is the Chapter 62-777 F.A.C. SCTL for soils (FDEP, 2005).

(2) The C5 to C7 range actually represents “post n-pentane” through C7 as a result of using pentane as the extraction solvent, hence quantitation of results begins at an area after C5 (n-pentane) and before C6 (n-hexane) and ends at C7.

FDEP Residential SCTL - FDEP Residential Direct Exposure under Chapter 62-777, F.A.C.
Leach to GW - SCTL - FDEP Leachability to Groundwater under Chapter 62-777, F.A.C.

Note that data will be reported at the LOQ and MDL, with non-detected data being the MDL followed by a “U” qualifier as per Florida state regulations. The LOD is presented for completeness and compliance with the DoD QSM, Version 4.1.

Bolded rows indicate that the PAL is between the laboratory LOQ and DL. The Project Team has agreed to accept this data for decision making if results below the LOQ are "J" qualified.

SAP Worksheet #16 -- Project Schedule / Timeline Table
(UFP-QAPP Manual Section 2.8.2)

Activities	Organization	Dates (MM/DD/YY) Anticipated Date(s) of Initiation	Dates (MM/DD/YY) Anticipated Date of Completion	Deliverable	Deliverable Due Date
Distribute Draft SAP for Navy Review	Tetra Tech	09/17/10	09/2410	Draft SAP	09/10/10
Navy Review of Draft SAP	Navy	09/27/10	10/15/10		
Revise SAP - Provide to FDEP for Review	Tetra Tech	10/18/10	10/29/10	Draft Final SAP	10/29/10
FDEP SAP Review/ Approval	FDEP	11/01/10	12/31/10		
Prepare Final SAP	Tetra Tech	01/03/11	01/07/11	Final SAP	01/07/11
Subcontractor Procurement/ Mobilization	Tetra Tech	01/03/11	01/28/11		
Field Activities	Tetra Tech	01/31/11	03/11/11		
Chemical Analysis	Tetra Tech	01/31/11	04/08/11		
Data Validation	Tetra Tech	03/25/11	04/29/11		
Prepare Draft Site Assessment Report (SAR) and Distribute for Navy Review	Tetra Tech	05/02/11	06/10/11	Draft SAR	06/10/11
Navy Review of Draft SAR	Navy	06/13/11	07/08/11		
Revise SAR - Provide to FDEP for Review	Tetra Tech	07/11/11	07/15/11	Draft Final SAR	07/15/11
FDEP SAP Review/ Approval	FDEP	07/18/11	09/06/11		
Prepare Final SAR	Tetra Tech	09/19/11	10/04/11	Final SAR	10/04/11

SAP Worksheet #17 -- Sampling Design and Rationale
(UFP-QAPP Manual Section 3.1.1)

To assess whether contamination is present at Site 5, subsurface soil and groundwater samples will be collected according to the ASP approved by FDEP, which was based on the requirements for closure of UST fuel pipelines and distribution systems containing Gasoline and Kerosene Analytical Groups. The ASP, along with the FDEP approval letter, is provided in Appendix A. According to this plan, a total of 90 subsurface soil samples will be collected at the following landmark locations:

- 1 soil sample at each bowser (55 total samples).
- 1 soil sample at each valve pit (4 total samples).
- 1 soil sample at each change in pipeline direction not associated with a valve pit (7 total samples).
- 1 soil sample at each joint between pipes of different diameter (4 total samples).
- 1 sample at the expansion loop (1 total sample).
- 1 sample at each bleeder (2 total samples).
- 1 sample for every 100 feet (approximately) of straight pipe for areas not enclosed in a service trench (17 total samples).

The locations of proposed soil samples are shown on Figure 17-1.

Sampling locations represent areas that were determined to be potential locations where fuel or oil may have been introduced to soil or groundwater. Bowsers were selected as sampling locations because they contained oil nozzles and tanks that may have leaked. In addition, fuel or oil may have been spilled at these locations during aircraft refueling or servicing. Valve pits, joints, elbows, and the expansion loop represent locations where welds would be located. Failure of such welds is a potential cause of leakage. Bleeder locations are a potential pathway for contaminants to enter soil or groundwater by nature of their function. Finally, a sample for every 100 feet of straight pipe was chosen to account for the potential for additional welded joints located along the run that are not depicted on design drawings. It is assumed that this spacing will enable any potential leak along the run to be detected.

Soil borings will be advanced no more 3 feet from the landmarks (i.e. bowsers, bleeders, valve pits, direction change, joint, and expansion loop) identified for sampling above. Soil samples for immediate FID screening will be collected at 2-foot vertical intervals beginning at 2 feet bgs and continuing below the estimated base of the landmark to 10 feet bgs. After these five samples have been collected at 2-foot intervals, additional samples will be collected at 5-foot intervals until two consecutive clean intervals (corrected FID readings of less than 10 ppm) are sampled, or until the top of the water table is encountered, which is estimated to be 40 bgs based on data from nearby monitoring wells.

If contamination is found, a UVF 3100 analyzer will be utilized on-site to screen step-out samples. Any step-out sample with a corrected FID measurement greater than 10 ppm will also be screened with the UVF analyzer.

One subsurface soil sample will be collected and submitted to Empirical for analysis at each of the 90 proposed soil borings. At least ten percent (or a minimum of 10 samples) of the step-out borings, a soil sample from the 2-foot interval with the highest UVF reading will be submitted to Empirical for analysis of the target analytes identified in Worksheet #15. If there are no UVF results because there were no FID readings > 10 ppm at a particular soil boring, then the 2-foot interval from just below the landmark (as determined by the Site Geologist) will be submitted to represent the potential source area at the location believed to be most likely to have contamination. Laboratory analytical results will be used to confirm FID and UVF screening data and to quantify the magnitude of contamination in areas where the highest concentrations of petroleum hydrocarbons are suspected.

To meet the requirements identified in Table B, Chapter 62-770, F.A.C., soil samples will be analyzed for VOCs, including BTEX and MTBE, using USEPA SW-846 Method 8260B; PAHs using USEPA SW-846 Method 3510C/3520 and 8270C; and TRPH using Florida Petroleum Range Organics (FL-PRO) method. If TRPH exceedances are detetected, TRPH speciation analysis will be performed via the TPHCWG Method on a minimum of 10 and maximum of 15 soil samples. A minimum of 10 samples is required so that upper confidence level (UCL) calculations can be performed. Samples for TRPH speciation will be selected by the Site Geologist in the field based on the UVF screening data with the intent to collect samples from locations exhibiting the highest on-site screening values by UVF for analysis by the off-site laboratory. This will allow for an evaluation of the type and distribution of aliphatic and aromatic petroleum hydrocarbons that remain in the soil in the SAR, if present. TRPH speciation is being performed because the FL-PRO method can provide false positive results when used for older petroleum sources that have degraded. Therefore, TRPH speciation will be performed to determine if any TRPH remaining at the site actually presents a risk to human health.

One groundwater sample will be collected and submitted to Empirical for analysis at every location where soil FID measurements exceed 10 ppm and the sample depth is within 20 feet of the water table. No groundwater sample is required to be collected if the distance between the water table and a soil FID exceedance (>10 ppm) is greater than 20 feet. However, a minimum of five groundwater samples will be collected, even if there are no soil FID measurements that exceed 10 ppm within 20 feet of the water table. The groundwater data is being collected because FDEP will not approve a NFA for a site without any groundwater data being collected. Assuming that there are no exceedances of soil FID measurements within 20 feet of the water table, the locations of the five (005, 014, 039, 049, and 084) proposed groundwater samples are dispersed across the Site 5 investigation area. See Figure 17-1 for sample locations. A groundwater DPT sample will be collected from the uppermost occurrence of groundwater (estimated to be 40 feet bgs) at the boring locations. These samples collected from the DPT screen point samplers will be analyzed for the petroleum related compounds specified in Table B Chapter 62-770, F.A.C., as identified in Worksheet #15.

If target analytes are identified in any of the 90 initially proposed DPT groundwater samples at concentrations that exceed FDEP GCTLs, step out samples will be collected to determine vertical and horizontal extent. The first step will be to vertically delineate the initial exceedance by performing vertical profiling in 10-foot increments. Once the vertical depth of contamination is determined, step-out groundwater samples will be collected 100 feet in each cardinal direction. Each step-out sample will have vertical profile samples collected down to the maximum depth reported for the sample location that required additional delineation. All groundwater samples will be submitted to an onsite mobile laboratory for analysis of BTEX and naphthalene compounds.

Following completion of the groundwater delineation, the Project Team will evaluate the data to the optimal location of a monitoring well network using existing site wells and up to 35 new monitoring wells. Once the new wells are installed, the monitoring well network will be sampled. To meet the requirements identified in Table B, Chapter 62-770, F.A.C. (summarized in Worksheet 15), groundwater monitoring well samples will be analyzed for VOCs, including BTEX, MTBE, 1,2-dichloroethane and other PPL VOHs using USEPA SW-846 Method 8260B; EDB using USEPA SW-846 Method 8011; PAHs using USEPA SW-846 Method 3510C/3520 and 8270C; TRPH using Florida Method FL-PRO; and total lead using USEPA SW-846 Method 3010A/6010C.

All data collected will undergo limited data validation, as described in Worksheet #36.

SAP Worksheet #18 -- Sampling Locations and Methods/SOP Requirements Table
(UFP-QAPP Manual Section 3.1.1)

Sampling Location/ID Number	Matrix	Depth (units)	Analytical Group	Number of Samples (identify field duplicates)	Sampling SOP Reference[1]
5-SS-01 to 5-SS-90 (Step-out borings will start with 5-SS-91 and continue numerically)	Soil	TBD	VOCs (BTEX and MTBE Only) PAHs TRPH	90 (plus 10 field duplicates)	FDEP SOPs FC1000, FD1000, FS1000, FS3000; Tetra Tech SOPs CT-04, SA-1.3, SA-6.1, SA-6.3, SA-7.1
TBD	Soil	TBD	TRPH Speciation[2]	10 (plus 1 field duplicate) (minimum of 10, maximum of 20)	FDEP SOPs FC1000, FD1000, FS1000, FS3000; Tetra Tech SOPs CT-04, SA-1.3, SA-6.1, SA-6.3, SA-7.1
5-MW-01 to 5-MW-TBD	Groundwater	TBD	VOCs EDB PAHs Total Lead TRPH	5 (minimum) (plus a minimum of 10% field duplicates)	FDEP SOPs FC1000, FD1000, FS1000, FS 2000, FS2200, FT1000, FT1100, FT1200, FT1400, FT1500, FT1600; Tetra Tech SOPs CT-04, SA-1.1, SA-6.1, SA-6.3, SA-7.1

1 SOP that describes the sample collection procedures, as identified in Worksheet #21.
2 TRPH Speciation will be analyzed on soil samples exhibiting the highest UVF and/or FID readings, which will ideally include all samples that exceed the TRPH PAL.

SAP Worksheet #19 -- Analytical SOP Requirements Table
(UFP-QAPP Manual Section 3.1.1)

Matrix	Analytical Group	Analytical and Preparation Method / SOP Reference[1]	Containers (number, size, and type)	Sample Volume (units)	Preservation Requirements (chemical, temperature, light protected)	Maximum Holding Time (preparation/ analysis)
Soil	VOCs (BTEX and MTBE Only)	SW-846 5035/8260B Empirical SOP202/225	Three 5-gram Encore samplers or terracores	5 grams	2x40 milliter (mL) in water and 1x40 mL in methanol, freeze to < -10 °C	48 hours from sampling to preparation, 14 days to analysis
	PAHs	SW-846 3546/8270C Empirical SOP201/343	One 4-ounce glass jar	15 grams	Cool to < 6 °C	14 days until extraction, 40 days to analysis
	TRPH	FL-PRO Empirical SOP338/343	One 4-ounce glass jar	15 grams	Cool to < 6 °C	14 days until extraction, 40 days to analysis
	TRPH Speciation	TPHCWG SunLabs TPHCWG Direct Method	One 4-ounce glass jar	10 grams	Cool to < 6 °C	14 days until extraction, 40 days to analysis
Groundwater and Aqueous Field QC Samples	VOCs	SW-846 5030/8260B Empirical SOP202	Three - 40 mL glass vials	5 mL	Hydrochloric acid (HCl) to pH<2; Cool to < 6 °C; no headspace	14 days to analysis
	EDB	SW-846 8011 Empirical SOP218	Three - 40 mL vials	40 mL	Cool to < 6 °C	14 days to analysis
	PAHs	SW-846 3510C/3520/8270C Empirical SOP201/300	Two 1 - liter (L) glass amber bottles	1,000 mL	Cool to < 6 °C	7 days until extraction, 40 days to analysis
	Total Lead	SW-846 3010A/6010C Empirical SOP100/105	One - 500 mL plastic bottle	50 mL / 30 mL mercury	Nitric acid (HNO_3) to pH <2; Cool to < 6 °C	180 days to analysis
	TRPH	FL-PRO Empirical SOP338	Two - 1L amber glass	1,000 mL	HCl to pH <2; Cool to < 6 °C	7 days until extraction, 40 days to analysis

Notes:

1 Laboratory SOPs are subject to revision and updates during duration of the project, the laboratory will use the most current revision of the SOP at the time of analysis.

SAP Worksheet #20 -- Field Quality Control Sample Summary Table

(UFP-QAPP Manual Section 3.1.1)

Matrix	Analytical Group	No. of Sampling Locations	No. of Field Duplicates	No. of MS/MSDs[1]	No. of Field Blanks[2]	No. of Equipment Blanks	No. of Trip Blanks	Total No. of Samples to Lab
Soil	VOCs (BTEX and MTBE Only)	90	10	5/5	0	5	5	110
	PAHs	90	10	5/5	0	5	NA	105
	TRPH	90	10	5/5	0	5	NA	105
	TRPH Speciation	10 (minimum)	1	1/1	0	1	NA	12
Groundwater	VOCs	5 (minimum)	1	1/1	0	1	1	8 (minimum)
	EDB	5 (minimum)	1	1/1	0	1	NA	7 (minimum)
	PAHs	5 (minimum)	1	1/1	0	1	NA	7 (minimum)
	Total Lead	5 (minimum)	1	1/1	0	1	NA	7 (minimum)
	TRPH	5 (minimum)	1	1/1	0	1	NA	7 (minimum)

1 Although the matrix spike/matrix spike duplicate (MS/MSD) are not typically considered a field QC and are not included in the "Total No. of Samples to Lab", they are included here because location determination is often established in the field.

SAP Worksheet #21 -- Project Sampling SOP References Table
(UFP-QAPP Manual Section 3.1.2)

Reference Number	Title, Revision Date and / or Number	Originating Organization of Sampling SOP	Equipment Type	Modified for Project Work? (Y/N)	Comments
CT-04	Sample Nomenclature (Revision 2, 3/09/09)	Tetra Tech	N/A	N	SOP contained in Appendix B.
CT-05	Database Records and Quality Assurance (Revision 2, 01/29/01)	Tetra Tech	N/A	N	SOP contained in Appendix B.
GH-1.5	Borehole and Sample Logging (Revision 1, June, 1999)	Tetra Tech	Rock Hammer, Knife, Camera, Dilute HCl, Ruler, Hand Lens	N	SOP contained in Appendix B.
GH-2.5	Groundwater Contour Maps and Flow Determinations (Revision 1, June, 1999)	Tetra Tech	N/A	N	SOP contained in Appendix B.
GH-2.8	Groundwater Monitoring Well Installation (Revision 3, September, 2003)	Tetra Tech	DPT and HSA combination drill rig	N	SOP contained in Appendix B.
SA-2.4	Soil Gas Sampling	Tetra Tech	FID	N	SOP contained in Appendix B.
SA-2.5	Direct Push Technology (Geoprobe®/Hydropunch™) (Revision3, 03/09/09)	Tetra Tech	DPT rigs	N	SOP contained in Appendix B.

Reference Number	Title, Revision Date and / or Number	Originating Organization of Sampling SOP	Equipment Type	Modified for Project Work? (Y/N)	Comments
HS-1.0	Utility Locating and Excavation Clearance (Revision 2, December, 2003)	Tetra Tech	Remote subsurface sensing, magnetometer, etc.	N	SOP contained in Appendix B.
SA-6.1	Non-Radiological Sample Handling, Revision 3, February 2004	Tetra Tech	Sample Bottle Ware, Packaging Material, Shipping Materials	N	SOP contained in Appendix B.
SA-6.3	Field Documentation Revision 3, 03/09/09)	Tetra Tech	Field Logbook, Field Sample Forms, Boring Logs	N	SOP contained in Appendix B.
SA-7.1	Decontamination of Field Equipment (Revision 6, 01/28/09)	Tetra Tech	Decontamination Equipment (scrub brushes, phosphate free detergent, DI water)	N	SOP contained in Appendix B.
SOP-05	Global Positioning System (Revision 1, July, 2010)	Tetra Tech	GPS unit	Y	SOP written specifically for this project. SOP contained in Appendix B.
FC-1000	Cleaning/Decontamination Procedures, December 2008	FDEP	Decontamination Equipment (scrub brushes, phosphate-free detergent, DI water)	N	SOP contained in Appendix B.
FD-4000	Documentation for Calibration of Field-Testing Instruments and Field Analysis	FDEP	Multi-parameter water quality meter, turbidity meter, and FID	N	SOP contained in Appendix B.
FQ-1310	Frequency	FDEP	Multi-parameter water quality meter, turbidity meter, and FID	N	SOP contained in Appendix B.
FS-1000	General Sampling, December 2008	FDEP	N/A	N	SOP contained in Appendix B.

Reference Number	Title, Revision Date and / or Number	Originating Organization of Sampling SOP	Equipment Type	Modified for Project Work? (Y/N)	Comments
FS-2000	General Water Sampling, December 2008	FDEP	N/A	N	SOP contained in Appendix B.
FS-2200	Groundwater Sampling, December 2008	FDEP	N/A	N	SOP contained in Appendix B.
FS-3000	Soil Sampling, December 2008	FDEP	Stainless steel auger bucket, extension rods, and T-handle	N	SOP contained in Appendix B.
FT-1000	General Field Testing and Measurement, December 2008	FDEP	Multi-parameter water quality meter, turbidity meter, and FID	N	SOP contained in Appendix B.
FT-1100	Field Measurement of Hydrogen Ion Activity (pH), December 2008	FDEP	Multi-parameter water quality meter	N	SOP contained in Appendix B.
FT-1200	Field Measurement of Specific Conductance (Conductivity), December 2008	FDEP	N/A	N	SOP contained in Appendix B.
FT-1400	Field Measurement of Temperature, December 2008	FDEP	Thermistor, thermometer, or temperature sensor	N	SOP contained in Appendix B.
FT-1500	Field Measurement of Dissolved Oxygen (DO), December 2008	FDEP	DO Sensor	N	SOP contained in Appendix B.
FT-1600	Field Measurement of Turbidity, December 2008	FDEP	Turbidity sensor	N	SOP contained in Appendix B.

Notes:

1 - FDEP Field SOPs can be obtained at the following website: http://www.dep.state.fl.us/labs/bars/sas/sop/index.htm.

SAP Worksheet #22 -- Field Equipment Calibration, Maintenance, Testing, and Inspection Table
(UFP-QAPP Manual Section 3.1.2.4)

Field Equipment	Activity[1]	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference[2]	Comments
DPT / Backhoe / Excavating Machinery	Inspection	Daily	Equipment Inspection Sheet Criteria	Operator correction or replacement	Tetra Tech FOL or designee	FS 3000, GA-1.5, SA-2.5	None
GPS	Positioning	Beginning and end of each day used	Accuracy: sub-meter horizontal dilution of precision < 3, number of satellites must be at least six	Wait for better signal, replace unit, or choose alternate location technique	Tetra Tech FOL or designee	SOP-05	None
Redi Flo™ Submersible Pump	Visual Inspection	Daily	Equipment Inspection Sheet Criteria	Operating correction or replacement	Tetra Tech FOL or designee	FS-2000	None
Water Quality Meter (YSI 600 Series or similar)	Visual Inspection Calibration/ Verification	Daily Beginning and end of day	Manufacturer's guidance	Operator correction or replacement	FOL or designee	GH-2.8, FS-2000, FT-1000 Series, Manufacturer's Guidance Manual, FT-1000.2., FQ-1310, and FD-4000	None
Turbidity Meter (LaMotte 2020 or equivalent)	Visual Inspection Calibration/ Verification	Daily Beginning and end of day	Manufacturer's guidance Calibrations must bracket expected values. Initial Calibration Verification (ICV) must be <5 NTU.	Operator correction or replacement	Tetra Tech FOL or designee	GH-2.8, FT-1600, Manufacturer's Guidance Manual, FT-1000.2., FQ-1310, and FD-4000	None

Field Equipment	Activity[1]	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference[2]	Comments
Electric Water Level Indicator	Visual Inspection Field checks as per manufacturer	Daily Once upon receiving from vendor	0.01 foot accuracy	Operator correction or replacement	Tetra Tech FOL or designee	FS-2000, Manufacturer's Guidance	None
FID	Visual Inspection Calibration/ Verification	Daily Beginning and end of day	Manufacturer's Guidance	Operator correction or replacement	Tetra Tech FOL or designee	Manufacturer's Guidance Manual, FT-1000.2., FQ-1310, and FD-4000	To be used to determine the subsurface soil depth that is most impacted for biased sample collection.
UVF 3100 Analyzer	Visual Inspection Calibration/ Verification	Daily Beginning and end of day	Manufacturer's Guidance	Operator correction or replacement	Tetra Tech FOL or designee	Manufacturer's Guidance Manual, FT-1000.2., FQ-1310, and FD-4000	To be used to determine the subsurface soil depth that is most impacted for biased sample collection.

1 Activities may include: calibration, verification, testing, and maintenance.
2 The appropriate reference letter or number from the Project Sampling SOP References Table (Worksheet #21).

SAP Worksheet #23 -- Analytical SOP References Table
(UFP-QAPP Manual Section 3.2.1)

Lab SOP Number	Title, Revision Date, and / or Number	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work?[1] (Y/N)
Empirical SOP100	Metals Digestion/ Preparation, Methods 3005A/ USEPA CLP ILMO 4.1 Aqueous, 3010A, 3030C, 3050B, USEPA CLP ILMO 4.1 (Soil/Sediment), 200.7, Standard Methods 3030C (Revision 20, 04/27/10)	Definitive	Groundwater and Aqueous Field QC Samples/ Lead Digestion	NA/ Preparation	Empirical	N
Empirical SOP105	Metals by Inductively Coupled Plasma-Atomic Emission Spectroscopy (ICP-AES) Technique, SW-846 Methods 6010B, 6010C, USEPA Method 200.7, Standard Methods 19th Edition 2340B, USEPA CLP ILMO 4.1 (Revision 16, 04/11/10)	Definitive	Groundwater and Aqueous Field QC Samples/ Lead	Inductively Coupled Plasma (ICP) – Atomic Emission Spectroscopy (AES)	Empirical	N
Empirical SOP201	GC/MS Semivolatiles and Low-Concentration PAHs by EPA Method 625 and SW846 Method 8270C and 8270D, Including Appendix IX Compounds (Revision 20, 04/26/10)	Definitive	Soil, Groundwater, and Aqueous Field QC Samples / Low Level PAHs	Gas Chromatography/ Mass Spectroscopy (GC/MS)	Empirical	N
Empirical SOP202	GC/MS Volatiles by EPA Method 624 and SW846 Method 8260B, Including Appendix IX Compounds (Revision 22, 09/30/09)	Definitive	Soil, Groundwater, and Aqueous Field QC Samples/ VOCs	GC/MS	Empirical	N
Empirical SOP218	GC/ECD 1,2-Dibromoethane (EDB) and 1,2-Dibromo-3-Chloropropane by EPA Methods 504.1 and SW-846 8011 (Revision 06, 09/30/09)	Definitive	Groundwater and Aqueous Field QC Samples / EDB	Gas Chromatography Electron Capture Detector (GC/ECD)	Empirical	N
Empirical SOP225	GC/MS Volatile Non-Aqueous Matrix Extraction Using SW-846 Method 5035 for 8260B Analysis (Revision 08, 09/24/08)	Definitive	Soil/ VOCs Extraction	GC/MS	Empirical	N

Lab SOP Number	Title, Revision Date, and / or Number	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work?[1] (Y/N)
Empirical SOP300	GC/MS- Semivolatile BNA-Aqueous Matrix Extraction Using SW-846 Method 3510C for 8270/625 Analysis (Revision 18, 04/26/10)	Definitive	Groundwater / PAHs Extraction	NA/ Extraction	Empirical	N
Empirical SOP338	FL-PRO (Extractable Petroleum Hydrocarbons) Aqueous and Solid Matrix (Revision 08, 04/29/10)	Definitive	Soil, Groundwater, and Aqueous Field QC Samples/ TRPH	GC/Flame Ionization Detection (FID)	Empirical	N
Empirical SOP343	BNA, Pesticides/PCB, and TPH Non-Aqueous Matrix Microwave Extraction Using SW-846 Method 3546 (Revision 18, 08/01/09)	Definitive	Soil/ PAH Extraction	NA/ Extraction	Empirical	N
Empirical SOP404	Laboratory Sample Receiving Log-in and Storage Standard Operating Procedures (Revision 13, 06/29/09)	NA	Log-in	NA/ Log-in	Empirical	N
Empirical SOP-405	Analytical Laboratory Waste Disposal (Revision 5, 06/23/09)	Definitive	Disposal	NA / Disposal	Empirical	N
Empirical SOP-410	Standard Operating Procedure (SOP) for Laboratory Sample Storage, Secure Areas, and Sample Custody (Revision 7, 06/23/09)	Definitive	Log-in	NA / Log-in	Empirical	N
SunLabs TPHCWG Direct Method	Method Manual for Total Petroleum Hydrocarbon Working Group TPHCWG Direct Method (Revision 0, 10/16/09)	Definitive	Groundwater, Soil, and Aqueous Field QC Samples / TRPH Speciation	GC/FID	SunLabs	N

Note:
Copies of all the Laboratory SOPs listed in this table are included in Appendix C.

SAP Worksheet #24 -- Analytical Instrument Calibration Table
(UFP-QAPP Manual Section 3.2.2)

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	SOP Reference[1]
GC/MS VOCs	Tune Verification - Bromofluorobenzene (BFB)	Prior to each Initial Calibration (ICAL) and at the beginning of each 12-hour period.	Must meet the ion abundance criteria required by the method (SW8260B; Section 7.3.1; Table 4).	Retune and/or clean or replace source. No samples may be accepted without a valid tune.	Analyst, Department Manager	Empirical SOP202
	ICAL – a minimum of a 5-point calibration is prepared for all target analytes	Upon instrument receipt, for major instrument changes, or when continuing calibration verification (CCV) does not meet criteria.	The average response factor (RF) for System Performance Check Compound (SPCCs) must be ≥ 0.30 for chlorobenzene and 1,1,2,2-tetrachloroethane, ≥ 0.1 for chloromethane, bromoform, and 1,1-dichloroethane. The percent relative standard deviation (%RSD) for RFs for calibration check compounds (CCCs) must be ≤ 30%; and %RSD for each target analyte must be ≤ 15%, or the linear regression correlation coefficient (r) must be ≥ 0.995; or the coefficient of determination (r^2) must be ≥ 0.99 (6 points are required for second order).	Correct problem then repeat ICAL. No samples may be run until ICAL has passed.	Analyst, Department Manager	
	Retention Time (RT) Window Position Establishment	Once per ICAL for each analyte and surrogate.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	NA.	Analyst, Department Manager	
	Evaluation of Relative Retention Times (RRTs)	With each sample.	RRT of each target analyte must be within ± 0.006 RRT units.	Correct problem, then rerun ICAL.		
	Initial Calibration Verification (ICV) – Second Source	Once after each ICAL, prior to beginning a sample run.	The percent recovery (%R) for all target analytes must be within 80-120% of true values.	Correct problem and verify ICV. If that fails, correct problem and repeat ICAL. No samples may be run until ICV has been verified.		

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	SOP Reference[1]
GC/MS VOCs	CCV	Perform one per 12-hour analysis period after tune and before sample analysis.	The minimum RF for SPCCs must be ≥ 0.30 for chlorobenzene and 1,1,2,2-tetrachloroethane, ≥ 0.1 for chloromethane, bromoform, and 1,1-dichloroethane. The percent difference or percent drift (%D) for all target analytes and surrogates must be ≤ 20%.	Correct problem and rerun CCV. If that fails, repeat ICAL and reanalyze all samples analyzed since the last successful CCV.	Analyst, Department Manager	Empirical SOP202
GC/MS PAHs	Tune Verification – decafluoro-triphenyl-phosphine (DFTPP)	At the beginning of each 12-hour analytical sequence.	Must meet the ion abundance criteria required by the method.	Retune and/or clean source.	Analyst, Department Manager	Empirical SOP201
	ICAL – a minimum of a 5-point calibration is prepared for all target analytes.	Instrument receipt, instrument change (new column, source cleaning, etc.), when CCV is out of criteria.	Average RF for SPCCs must be ≥0.050; %RSD for RFs for CCCs must be < 15% for all compounds. If not met: Option 1) r must be ≥ 0.995. Option 2) r^2 must be ≥ 0.99 (6 points for second order).	Recalibrate and/or perform the necessary equipment maintenance. Check the calibration standards. Reanalyze the affected data.	Analyst, Department Manager	
	ICV – Second Source	Once after each ICAL prior to beginning a sample run.	%R of each analyte must be within 80-120% of true value. SPCC RFs must be ≥ 0.050. CCCs must be ≤ 20%D.	Identify source of problem, correct, repeat calibration, rerun samples.	Analyst, Department Manager	
	RT Window Position Establishment	Once per ICAL for each analyte and surrogate.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	NA.	Analyst, Department Manager	
	Evaluation of RRTs	With each sample.	RRT of each target analyte must be within ± 0.006 RRT units.	Correct problem, then rerun ICAL.	Analyst, Department Manager	
	CCV	Analyze a standard at the beginning of each 12-hour shift after a DFTPP tune.	%D for all target compounds must be ≤ 20%; SPCC RFs must be ≥0.050.	Recalibrate and/or perform the necessary equipment maintenance. Check the calibration standards. Reanalyze the affected data.	Analyst, Department Manager	

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	SOP Reference[1]
GC/FID TRPH	ICAL – a minimum of a 5-point calibration is prepared for all target analytes.	Perform after major instrument maintenance and upon failure of second consecutive CCV, prior to sample analysis.	The %RSD for each analyte must be ≤ 20%, If not met: Option 1) r must be ≥ 0.995. Option 2) r^2 must be ≥ 0.99 (6 points for second order).	Recalibrate and/or perform the necessary equipment maintenance. Check the calibration standards. Reanalyze the affected data.	Analyst, Department Manager	Empirical SOP338
	ICV – Second Source	After each ICAL.	The %R must be within 75-125% of the true value.	Determine problem and Recalibrate.	Analyst, Department Manager	
	CCV	At the beginning of a sequence and after every 12 hours or 10 samples (whichever comes first), then at the end of the sequence.	The %R must be within 75-125% of the true value.	If the CCV fails high, report samples that are less than the LOQ. Recalibrate and/or reanalyze samples back to last acceptable CCV.	Analyst, Department Manager	

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	SOP Reference[1]
ICP- AES Total Lead	ICAL - the instrument is calibrated by a 1-point calibration per manufacturer's guidelines.	At the beginning of each day, or if the QC is out of criteria.	None; only one high standard and a calibration blank must be analyzed. If more than one calibration standard is used, r must be ≥ 0.995.	Recalibrate and/or perform the necessary equipment maintenance. Check the calibration standards. Reanalyze the affected data.	Analyst, Department Manager	Empirical SOP100/105
	ICV – Second Source	Following ICAL, prior to the analysis of samples.	The %R must be within 90-110% of the true value.	Investigate reasons for failure, reanalyze once. If still unacceptable, repeat calibration.	Analyst, Department Manager	
	Initial Calibration Blank (ICB)	Before beginning a sample sequence.	No analytes detected > LOD.	Correct the problem, then re-prepare and reanalyze.	Analyst, Department Manager	
	CCV	Analyze a standard at the beginning and end of the sequence and after every 10 samples.	The %R must be within 90-110% of true value.	Recalibrate and/or perform the necessary equipment maintenance. Check the calibration standards. Reanalyze the affected data.	Analyst, Department Manager	
	Continuing Calibration Blank (CCB)	After the initial CCV, after every 10 samples, and at the end of the sequence.	No analytes detected > LOD.	Correct the problem, then re-prepare and reanalyze calibration blank and previous 10 samples.	Analyst, Department Manager	
	Low-Level Check Standard	Daily after ICAL and before samples.	The %R must be within 80-120% of the true value.	Investigate and perform necessary equipment maintenance. Recalibrate and reanalyze all affected samples.	Analyst, Department Manager	
	Interference Check Standards (ICS – ICS A and ICS B)	At the beginning and end of an analytical run and after each batch of 20 samples.	The absolute value of ICS A recoveries for non-spiked analytes must be ≤ LOD; and ICS B %Rs must be within 80-120% of the true value.	Investigate and perform necessary equipment maintenance. Recalibrate and reanalyze all affected samples.	Analyst, Department Manager	

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	SOP Reference[1]
GC/ECD EDB	ICAL - A minimum 5 point calibration is required.	Calibrate the instrument when it is received and after a major change or if the daily calibration fails.	The %RSD must be ≤20%, or r must be ≥ 0.995, or r^2 must be ≥ 0.99 (minimum of 6 points required for second order).	Recalibrate and/or perform necessary equipment maintenance. Check calibration standards. Reanalyze affected data.	Analyst	Empirical SOP218
	ICV	Once after each initial calibration.	The %R of the target analyte must be within 80-120% of true value.	Identify source of problem, correct, repeat calibration, rerun samples	Analyst	
	CCV	Analyze standard at the beginning and end of sequence and every 10 field samples.	The %D of the target analyte must be ≤20%.	If %D is high and sample result is ND (non detect), qualify/narrate with project approval. If %D is low or project approval not received, reanalyze all samples since the last successful CCV.	Analyst	

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	SOP Reference[1]
GC/FID TRPH Speciation	ICAL – A minimum 5 point calibration is required.	Perform after major instrument maintenance and upon failure of second consecutive CCV, prior to sample analysis.	The %RSD for each analyte must be ≤ 25%, If not met: Option 1) r must be ≥ 0.995. Option 2) r^2 must be ≥ 0.99 (6 points for second order).	Recalibrate and/or perform the necessary equipment maintenance. Check the calibration standards. Reanalyze the affected data.	Analyst, Department Manager	SunLabs TPHCWG Direct Method
	ICV	After each ICAL, prior to sample analysis.	The %R must be within 75-125% of the true value.	Determine problem and Recalibrate.	Analyst, Department Manager	
	CCV	Prior to sample analysis and every 10 samples.	The %D must be within 75-125% of the true value.	If the CCV fails high, report samples that are less than the LOQ. Recalibrate and/or reanalyze samples back to last acceptable CCV.	Analyst, Department Manager	

SAP Worksheet #25 -- Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table

(UFP-QAPP Manual Section 3.2.3)

Instrument / Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person[2]	SOP Reference[1]
GC/MS	Check pressure and gas supply daily. Bake out trap and column, manual tune if BFB not in criteria, change septa as needed, cut column as needed, change trap as needed. Other maintenance specified in lab Equipment Maintenance SOP.	VOCs	Ion source, injector liner, column, column flow, purge lines, purge flow, trap.	Prior to ICAL and/or as necessary.	Acceptable ICAL and CCV.	Correct the problem and repeat ICAL or CCV.	Analyst, Department Manager	Empirical SOP202
GC/MS	Check pressure and gas supply daily. Manual tune if DFTPP not in criteria, change septa as needed, change liner as needed, cut column as needed. Other maintenance specified in lab Equipment Maintenance SOP.	PAHs	Ion source, injector liner, column, column flow.	Prior to ICAL and/or as necessary.	Acceptable ICAL and CCV.	Correct the problem and repeat ICAL or CCV.	Analyst, Department Manager	Empirical SOP201
ICP-AES	Clean torch assembly and spray chamber when discolored or when degradation in data quality is observed. Clean nebulizer, check argon, replace peristaltic pump tubing as needed. Other maintenance specified in lab Equipment Maintenance SOP.	Total Lead	Torch, nebulizer chamber, pump, pump tubing.	Prior to ICAL and as necessary.	Acceptable ICAL and CCV.	Correct the problem and repeat ICAL or CCV.	Analyst, Department Manager	Empirical SOP100/105
GC/ECD	Check pressure and gas supply daily. Change septa and/or liner as needed, replace or cut column as needed. Other maintenance specified in lab Equipment Maintenance SOP.	EDB	Injector liner, septa, column, column flow.	Prior to ICAL and/or as necessary.	Acceptable ICAL and CCV.	Correct the problem and repeat ICAL or CCV.	Analyst, Department Manager	Empirical SOP218

Instrument / Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person[2]	SOP Reference[1]
GC/FID	Check pressure and gas supply daily. Change septa and/or liner as needed, replace or cut column as needed. Check flame and FID jet. Other maintenance specified in lab Equipment Maintenance SOP.	TRPH	Injector liner, septa, column, column flow, flame, jet	Prior to ICAL and/or as necessary.	Acceptable ICAL and CCV.	Correct the problem and repeat ICAL or CCV.	Analyst, Department Manager	Empirical SOP338
GC/FID	Check pressure and gas supply daily. Change septa and/or liner as needed, replace or cut column as needed. Other maintenance specified in lab Equipment Maintenance SOP.	TRPH Speciation	Injector liner, septa, column, column flow.	Prior to ICAL and/or as necessary.	Acceptable ICAL and CCV.	Correct the problem and repeat ICAL or CCV.	Analyst, Department Manager	SunLabs TPHCWG Direct Method

SAP Worksheet #26 -- Sample Handling System
(UFP-QAPP Manual Appendix A)

Sample Collection, Packaging, and Shipment
Sample Collection (Personnel/Organization): FOL or designee/ Tetra Tech
Sample Packaging (Personnel/Organization): FOL or designee/ Tetra Tech
Coordination of Shipment (Personnel/Organization): FOL or designee/ Tetra Tech
Type of Shipment/Carrier: Federal Express
Sample Receipt and Analysis
Sample Receipt (Personnel/Organization): Sample Custodians/ Empirical, Sun Labs
Sample Custody and Storage (Personnel/Organization): Sample Custodians/ Empirical, Sun Labs
Sample Preparation (Personnel/Organization): Extraction Lab, Metals Preparation Lab/ Empirical, Sun Labs
Sample Determinative Analysis (Personnel/Organization): Gas Chromatography Lab, Gas Chromatography/Mass Spectrometry Lab, Metals Lab/ Empirical, Sun Labs
Sample Archiving
Field Sample Storage (No. of days from sample collection): 60 days from receipt
Sample Extract/ Digestate Storage (No. of days from extraction/digestion): 3 months from sample digestion/extraction
Biological Sample Storage (No. of days from sample collection): NA
Sample Disposal
Personnel/Organization: Sample Custodians/ Empirical, Sun Labs

SAP Worksheet #27 – Sample Custody Requirements Table
(UFP-QAPP Manual Section 3.3.3)

27.1 FIELD SAMPLE CUSTODY PROCEDURES

Sample Chain-of-Custody forms will be completed per Tetra Tech SOP SA-6.3. An example is included in Appendix B.

The following sections outline the procedures that will be used to document project activities and sample collection, handling tracking, and custody procedures during the investigation.

27.2 SAMPLE NOMENCLATURE

Worksheet #18 presents the sample nomenclature for the field and lists QA/QC samples to be collected.

27.3 SAMPLE COLLECTION AND DOCUMENTATION

Documentation of field observations will be recorded in a field logbook and/or on field log sheets including sample collection logs and boring logs. Bound, water-resistant field logbooks will be used for this project. Logbook pages will be numbered sequentially, and observations will be recorded with indelible ink.

Field sample log sheets will be used to document sample collection details. Other observations and activities will be recorded in the field logbook. Daily instrument calibration will be recorded in instrument calibration logs. Example field forms are included in Appendix B.

For sampling and field activities, the following types of information will be recorded in the field logbook, as appropriate:

- Site name and location
- Date and time
- Personnel and their affiliations
- Weather conditions
- Activities associated with sampling
- Subcontractor activity summary
- Site observations including site entry and exit times
- Site sketches monitoring well layout, if different than sampling plan figures
- Visitor names, affiliations, and arrival and departure times
- Health and safety issues including PPE

27.4 SAMPLE PACKAGING AND SHIPPING

Samples will be prepared for shipping using the following guidelines:

- Place properly identified sample container, with lid securely fastened, in a plastic bag (i.e., Ziploc-type bag), and seal bag.
- Place sample in a sturdy cooler that has been lined with a large plastic bag (i.e., garbage bag). Drain plugs on coolers should be taped shut.
- Place a temperature check indicator provided by the laboratory in each cooler to be shipped.
- Pack with sufficient cushioning materials, such as bubble wrap, to minimize the possibility of the container breaking.
- If cooling is required, pack sample containers in ice to adequately cool sample to 0 to 6 °C.
- Seal large liner bag by taping or knotting open end.
- Tape the original top, signed copy of the chain-of-custody form shall be placed in a large Ziploc-type bag inside the lid of the shipping cooler. If multiple coolers are sent but samples are included on one chain-of-custody form, the chain-of-custody form should then indicate how many coolers are included with the shipment.
- Close and seal the outside of shipping cooler using strapping tape. Place custody seals across the lid and body of cooler and under strapping tape to prevent tampering while in transit. No Department of Transportation (DOT) marking is required.

27.5 SAMPLE HANDLING AND TRACKING SYSTEM

Sample handling is described in Worksheet #26. Samples must be delivered to the laboratory via a public courier (e.g., Federal Express). Samples must be sent to the laboratory within 24 hours of being collected. Under no circumstances should sample holding times be exceeded.

27.6 SAMPLE CUSTODY

To ensure the integrity of a sample from collection through analysis, it is necessary to have an accurate written record that traces the possession and handling of the sample. This documentation is referred to as the chain-of-custody form. The chain of custody begins at the time of sample collection. The laboratory will provide forms that will be used for chain-of-custody documentation.

A sample is under custody if:

- The sample is in the physical possession of an authorized person;
- The sample is in view of an authorized person after being in his/her possession;
- The sample is placed in a secure area by an authorized person after being in his/her possession;
- The sample is in a secure area, restricted to authorized personnel only.

Custody documentation is designed to provide documentation of preparation, handling, storage, and shipping of all samples collected. A multi-part form is used. Each page of the form is signed and dated by the recipient of a sample or portion of sample. The person releasing the sample and the person receiving the sample will each retain a copy of the form each time a sample transfer occurs.

Integrity of the samples collected during the site investigation will be the responsibility of identified persons from the time the samples are collected until the samples, or their derived data, are incorporated into the analytical report.

The Tetra Tech FOL is responsible for the care and custody of the samples collected until they are delivered to the laboratory or are entrusted to a shipping courier. When transferring samples, the individuals relinquishing and receiving the samples will each sign the chain-of-custody form. The date and time will be recorded to each time the samples change hands. Once delivered to the laboratory, internal sample custody procedures will be followed as defined in the laboratory SOPs included in Appendix C.

27.6.1 <u>Field Sampling Custody Requirements</u>

Field Sample Custody Procedures (sample collections, packaging, and shipping to laboratory) will be conducted according to Tetra Tech SOP SA-6.3 (Appendix B). Following sample collections in the appropriate bottle ware, all samples will be immediately placed on ice in a cooler. The glass sample containers will be enclosed in bubble wrap to protect the bottle ware during shipment and to prevent cross contamination should a bottle break in transit. The cooler will be secured using duct tape or clear packaging tape along with two signed custody seals. Sample coolers will be delivered to a local courier location for priority overnight delivery to the selected laboratory for analysis.

The Tetra Tech FOL is responsible for the care and custody of the samples until they are delivered to the laboratory or are entrusted to a carrier. When transferring samples, the individuals relinquishing and receiving them will sign, date, and note the time on the chain-of-custody form. This form documents the sample custody transfer from the sampler to the laboratory, often through another person or agency (common carrier).

27.6.2 Laboratory

Laboratory sample custody procedures (receipt of samples, archiving, and disposal) will be used according to Empirical SOPs (Appendix C). Coolers are received and checked for proper temperature. A sample cooler receipt form will be filled out to note conditions and any discrepancies. The chain-of-custody form will be checked against the sample containers for accuracy. Samples will be logged into the Laboratory Information Management System (LIMS) and given a unique log number which can be tracked through processing. The Laboratory PM will notify the Tetra Tech FOL verbally or via e-mail of any problems on the same day that an issue is identified.

SAP Worksheet #28 -- Laboratory QC Samples Table (UFP-QAPP Manual Section 3.4)

Matrix	Soil, Groundwater, and Aqueous Field QC Samples
Analytical Group	VOCs
Analytical Method/SOP Reference	SW-846 8260B Empirical SOP202

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	One per preparation batch of 20 or fewer samples of similar matrix.	No analytes > ½ LOQ, except common lab contaminants, which must be < LOQ.	Investigate source of contamination. Rerun method blank prior to analysis of samples if possible. Evaluate the samples and associated QC: if blank results are above LOQ, then report sample results which are < LOQ or > 10X the blank concentration. Reanalyze blank and samples >LOQ and < 10X the blank.	Analyst, Laboratory Department Manager, and Data Validator	Bias/ Contamination	Same as Method/SOP QC Acceptance Limits.
Surrogate	Four per sample: Dibromofluoromethane 1,2-dichloroethane-d4 Toluene-d8 BFB	%Rs must meet the DoD QSM Version 4.1 limits as per Appendix G of the DoD QSM.	If sample volume is available, then re-prepare and reanalyze for confirmation of matrix interference when appropriate.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/ Bias	Same as Method/SOP QC Acceptance Limits.
Matrix Spike/ Matrix Spike Duplicate (MS/MSD)	One per sample delivery group (SDG) or every 20 samples of similar matrix.	%Rs must meet the DoD QSM Version 4.1 limits as per Appendix G of the DoD QSM. The RPD between MS and MSD should be ≤ 30%.	Corrective actions will not be taken for samples when recoveries are outside limits and surrogate and LCS criteria are met. If both the LCS and MS/MSD %Rs are unacceptable, then re-prepare the samples and QC.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/ Bias/ Precision	Same as Method/SOP QC Acceptance Limits.

Matrix	Soil, Groundwater, and Aqueous Field QC Samples
Analytical Group	VOCs
Analytical Method/SOP Reference	SW-846 8260B Empirical SOP202

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Laboratory Control Sample (LCS) / Laboratory Control Sample Duplicate (LCSD) (not required)	One is performed for each batch of up to 20 samples.	%Rs must meet the DoD QSM Version 4.1 limits as per Appendix G of the DoD QSM. If LCSD performed - The RPD between LCS and LCSD must be ≤ 30%.	Evaluate and reanalyze if possible. If an MS/MSD was performed in the same 12 hour clock and acceptable, then narrate. If the LCS %Rs are high, but the sample results are <LOQ, then narrate. Otherwise, re-prepare and reanalyze.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/ Bias Precision also, if LCSD is analyzed	Same as Method/SOP QC Acceptance Limits.
Internal Standard (IS)	Three per sample- Fluorobenzene Chlorobenzene-d5 1,4-dichlorobezene-d4	RTs for ISs must be within ± 30 seconds and the response areas must be within -50% to +100% of the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	Inspect mass spectrometer or gas chromatograph for malfunctions; mandatory reanalysis of samples analyzed while system was malfunctioning.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/ Bias	Same as Method/SOP QC Acceptance Limits.
Results between DL and LOQ	NA	Apply “J” qualifier to results detected between DL and LOQ.	NA.	Analyst, Supervisor, Data Validator	Accuracy	Same as QC Acceptance Limits.

Matrix	Soil, Groundwater, and Aqueous Field QC Samples
Analytical Group	PAHs
Analytical Method/SOP Reference	SW-846 8270C Empirical SOP201

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	One per preparation batch of 20 or fewer samples of similar matrix.	No target compounds > ½ the LOQ.	(1) Investigate source of contamination (2) Re-prepare and analyze method blank and all samples processed with the contaminated blank.	Analyst, Laboratory Department Manager and Data Validator	Bias/ Contamination	Same as Method/SOP QC Acceptance Limits.
Surrogates	Two per sample: 2-Fluorobiphenyl Terphenyl-d14	%Rs must meet the laboratory limits of 34-167 for waters and 14-129 for soils.	(1) Check chromatogram for interference; if found, then flag data. (2) If not found, then check instrument performance; if problem is found, then correct and reanalyze. (3) If still out, then re-extract and analyze sample. (4) If reanalysis is out, then flag data.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/ Bias	Same as Method/SOP QC Acceptance Limits.
MS/MSD	One per SDG or every 20 samples of similar matrix.	%Rs should meet the laboratory limits provided in Appendix C.[1] RPD between MS and MSD should be ≤ 30%.	Corrective Action will not be taken for samples when %Rs are outside limits and surrogate and LCS criteria are met. If both the LCS and MS/MSD are unacceptable, then re-prepare the samples and QC.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/ Bias / Precision	Same as Method/SOP QC Acceptance Limits.

Matrix	Soil, Groundwater, and Aqueous Field QC Samples
Analytical Group	PAHs
Analytical Method/SOP Reference	SW-846 8270C Empirical SOP201

QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
LCS LCSD (not required)	One is performed for each batch of up to 20 samples.	%Rs must meet the laboratory limits provided in Appendix C.[1] If LCSD performed - The RPD between LCS and LCSD must be ≤ 30%.	Evaluate and reanalyze if possible. If an MS/MSD was performed in the same 12 hour clock and is acceptable, then narrate. If the LCS recoveries are high but the sample results are <LOQ, then narrate. Otherwise, re-prepare and reanalyze.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy / Bias/ Precision also, if LCSD is analyzed	Same as Method/SOP QC Acceptance Limits.
IS	Two per sample – Phenanthrene-d10 Perylene-d12	RTs for ISs must be within ± 30 seconds and the response areas must be within -50% to +100% of the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	Reanalyze affected samples.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/ Bias	Same as Method/SOP QC Acceptance Limits.
Results between DL and LOQ	NA	Apply “J” qualifier to results detected between DL and LOQ.	NA.	Analyst, Supervisor, Data Validator	Accuracy	Same as QC Acceptance Limits.

1 Please note that laboratory derived limits are updated periodically and may change from the issuance of the final SAP to the time data validation is performed. The limits used for validation will be the limits that are current at the time of analysis.

Matrix	Groundwater and Aqueous Field QC Samples
Analytical Group	Total Lead
Analytical Method/SOP Reference	SW-846 6010C Empirical SOP100/105

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	One per digestion batch of 20 or fewer samples.	No analytes detected > ½ the LOQ.	If the blank value > LOQ, then report sample results. If the blank value < LOQ or > 10x the blank value; then redigest. If blank value is less than negative LOQ, then report sample results. If > 10x the absolute value of the blank result, then redigest.	Analyst, Laboratory Department Manager, and Data Validator	Bias/ Contamination	Same as Method/SOP QC Acceptance Limits.
LCS	One is performed for each batch of up to 20 samples.	The %R must be within 80-120%.	Redigest and reanalyze all associated samples for affected analyte.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/ Bias	Same as Method/SOP QC Acceptance Limits.
MS	One per preparation batch of 20 or fewer samples of similar matrix.	%R should be within 80-120% of true value (if sample is < 4x spike added).	Flag results for affected analytes for all associated samples with "N".	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/ Bias	Same as Method/SOP QC Acceptance Limits.
Duplicate Sample	One per preparation batch of 20 or fewer samples of similar matrix.	The RPD should be within ≤20%.	Narrate any results that are outside control limits.	Analyst, Laboratory Department Manager, and Data Validator	Precision	Same as Method/SOP QC Acceptance Limits.

Matrix	Groundwater and Aqueous Field QC Samples
Analytical Group	Total Lead
Analytical Method/SOP Reference	SW-846 6010C Empirical SOP100/105

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Serial Dilution	One is performed for each preparation batch with sample concentration(s) > 50x LOQ.	The 5-fold dilution result must agree within ±10%D of the original sample result if result is >50x LOD.	Perform Post Digestion Spike.	Analyst, Laboratory Department Manager, and Data Validator	Precision	Same as Method/SOP QC Acceptance Limits.
Post Digestion Spike	One is performed when serial dilution fails or target analyte concentration(s) in all samples are < 50x LOD.	The %R must be within 75-125% of expected value to verify the absence of an interference. Spike addition should produce a concentration of 10-100x LOQ.	Flag results for affected analytes for all associated samples with “J”.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/ Bias	Same as Method/SOP QC Acceptance Limits.
Results between DL and LOQ	NA.	Apply “J” qualifier to results between DL and LOQ.	NA.	Analyst, Supervisor, Data Validator	Accuracy	Same as QC Acceptance Limits.

Matrix	Soil, Groundwater, and Aqueous Field QC Samples
Analytical Group	TRPH
Analytical Method/SOP Reference	FL-PRO Empirical SOP338

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	One per preparation batch of 20 or fewer samples of similar matrix.	Must be ≤1/2 the LOQ.	Re-clean, retest, re-extract, reanalyze, and/or qualify the data.	Analyst, Laboratory Supervisor and Data Validator	Bias / Contamination	Same as Method/SOP QC Acceptance Limits.
Surrogates	2 per sample: 2-Fluorobiphenyl o-Terphenyl	2- Fluorobiphenyl - %Rs must meet the laboratory limits of 34-167 for waters and 14-129 for soils. o-Terphenyl - %Rs must meet the laboratory limits of 50-100 for waters and soils.	(1) Prepare again and reanalyze for confirmation of matrix interference when appropriate.	Analyst, Laboratory Supervisor and Data Validator	Accuracy /Bias	Same as Method/SOP QC Acceptance Limits.
LCS	One per preparation batch of 20 or fewer samples of similar matrix.	Water 55-118%R Soil 63-143%R If LCSD performed - The RPD between LCS and LCSD must be ≤ 20% (water) and ≤ 25% (soil).	(1) Evaluate and reanalyze if possible. (2) If an MS/MSD was performed in the same 12 hour clock and acceptable, then narrate. (3) If the LCS recoveries are high but the sample results are <LOQ, then narrate. Otherwise prepare again and reanalyze the batch.	Analyst, Laboratory Supervisor and Data Validator	Accuracy / Bias	Same as Method/SOP QC Acceptance Limits.
MS/MSD	One per SDG or every 20 samples of similar matrix.	Water 41-100%R Soil 51-215%R RPD between MS and MSD should be ≤ 20% (water) and ≤ 25% (soil).	(1) Corrective action will not be taken for samples when recoveries are outside limits and surrogate and LCS criteria are met. (2) If both the LCS and MS/MSD are unacceptable, then re-prepare the samples again and QC.	Analyst, Laboratory Supervisor and Data Validator	Precision / Accuracy / Bias	Same as Method/SOP QC Acceptance Limits.

1 Please note that laboratory derived limits are updated periodically and may change from the issuance of the final SAP to the time data validation is performed. The limits used for validation will be the limits that are current at the time of analysis.

Matrix	Groundwater and Aqueous Field QC Samples
Analytical Group	EDB
Analytical Method/SOP Reference	SW-846 8011 Empirical SOP218

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	One per preparatory batch of 20 or fewer samples of similar matrix.	All target analytes must be ≤ ½ LOQ.	Investigate source of contamination. Evaluate the samples and associated QC: i.e., if the blank results are above the LOQ, then report sample results that are <LOQ or > 10X the blank concentration. Otherwise, re-prepare a blank and samples >LOQ and <10X LOQ.	Analyst, Supervisor, Data Validator	Bias/ Contamination	Same as QC Acceptance Limits.
LCS	One per preparatory batch of 20 or fewer samples of similar matrix.	The %R must be within 70%-130% of true value. If LCSD performed - The RPD between LCS and LCSD must be ≤ 30%.	If an MS/MSD was performed and is acceptable, then narrate. If an LCS/ LCSD were performed and only one of the set was unacceptable, then narrate. If the LCS recovery is high, but the sample results are <LOQ, then narrate. Otherwise, re-extract blank and affected sample batch.	Analyst, Supervisor, Data Validator	Accuracy/ Bias	Same as QC Acceptance Limits.
MS/MSD	One per preparatory batch of 20 or fewer samples of similar matrix. (spike same as LCS)	The %R must be within 70%-130% of true value (if sample is < 4x spike added). RPD between MS and MSD should be ≤ 30%.	Evaluate the samples and associated QC and if the LCS results are acceptable, then narrate. If both the LCS and MS/MSD are unacceptable, then re-prepare the samples and QC.	Analyst, Supervisor, Data Validator	Accuracy/ Bias/ Precision	Same as QC Acceptance Limits.

Matrix	Groundwater and Aqueous Field QC Samples
Analytical Group	EDB
Analytical Method/SOP Reference	SW-846 8011 Empirical SOP218

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Second Column Confirmation	All positive results must be confirmed.	Results between primary and second column must be RPD ≤ 40%. Report the higher of the two concentrations, unless there is interference.	None. Apply qualifier if RPD >40% and discuss in the case narrative.	Analyst, Supervisor, Data Validator	Accuracy	Same as QC Acceptance Limits.
Results between DL and LOQ	NA	Apply “J” qualifier to results between DL and LOQ.	NA	Analyst, Supervisor, Data Validator	Accuracy	Same as QC Acceptance Limits.

1 Please note that laboratory derived limits are updated periodically and may change from the issuance of the final SAP to the time data validation is performed. The limits used for validation will be the limits that are current at the time of analysis.

Matrix	Soil
Analytical Group	TRPH Speciation
Analytical Method/SOP Reference	TPHCWG Method SunLabs TPHCWG Direct Method

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	One per preparation batch of 20 or fewer samples of similar matrix.	Must be <1/2 the LOQ.	Re-clean, retest, re-extract, reanalyze, and/or qualify the data.	Analyst, Laboratory Supervisor and Data Validator	Bias / Contamination	Same as Method/SOP QC Acceptance Limits.
LCS	One per preparation batch of 20 or fewer samples of similar matrix.	The %R must be within 60-140%. If LCSD performed - The RPD between LCS and LCSD must be ≤ 30%.	(1) Evaluate and reanalyze if possible. (2) If an MS/MSD was performed in the same 12 hour clock and acceptable, then narrate. (3) If the LCS recoveries are high but the sample results are <LOQ, then narrate. Otherwise prepare again and reanalyze the batch.	Analyst, Laboratory Supervisor and Data Validator	Accuracy / Bias	Same as Method/SOP QC Acceptance Limits.
MS/MSD	One per SDG or every 20 samples of similar matrix.	The %R should be within 60-140%. RPD between MS and MSD should be ≤ 30%.	(1) Corrective action will not be taken for samples when recoveries are outside limits and surrogate and LCS criteria are met. (2) If both the LCS and MS/MSD are unacceptable, then re-prepare the samples again and QC.	Analyst, Laboratory Supervisor and Data Validator	Precision / Accuracy /Bias	Same as Method/SOP QC Acceptance Limits.

1 Please note that laboratory derived limits are updated periodically and may change from the issuance of the final SAP to the time data validation is performed. The limits used for validation will be the limits that are current at the time of analysis.

SAP Worksheet #29 -- Project Documents and Records Table
(UFP-QAPP Manual Section 3.5.1)

Document	Where Maintained
Sample Collection Documents and Records: • Field logbook (and sampling notes) • Field sample forms (e.g., boring logs, sample logsheets, drilling logs, etc.) • Chain-of-custody records • Sample shipment airbills • Equipment calibration logs • Photographs • Field task modification forms • Sampling and analysis plan • Field Sampling SOPs	Tetra Tech project file; results will be provided in an Investigation Report.
Laboratory Documents and Records: • Sample receipt/login form • Equipment calibration logs • Sample analysis run logs • CA forms • Reported results for standards, QC checks, and QC samples • Data completeness checklists • Raw data	Tetra Tech project file; long-term data package storage at third party commercial document storage firm.
Data Assessment Documents and Records: • Field Sampling Audit Checklist (if an audit is conducted) • Analytical Audit Checklist (if an audit is conducted) • Data Validation Memoranda • All Versions of Project Reports	Tetra Tech project file.

SAP Worksheet #30 -- Analytical Services Table
(UFP-QAPP Manual Section 3.5.2.3)

Matrix	Analytical Group	Sample Locations/ID Number	Analytical Method	Data Package Turnaround Time	Laboratory / Organization[1] (name and address, contact person and telephone number)	Backup Laboratory / Organization[1] (name and address, contact person and telephone number)
Groundwater and Aqueous Field QC Samples	VOCs	See Worksheet #18	SW-846 8260B	21 calendar days	Kim Kostzer Empirical Laboratories, LLC 621 Mainstream Drive, Suite 270 Nashville, TN 37228 615-345-1115	NA
	PAHs	See Worksheet #18	SW-846 8270C			
	Total Lead	See Worksheet #18	SW-846 6010C			
	TRPH	See Worksheet #18	FDEP FL-PRO			
	EDB	See Worksheet #18	SW-846 8011			
Soil	VOCs (BTEX and MTBE Only)	See Worksheet #18	SW-846 8260B			
	PAHs	See Worksheet #18	SW-846 8270C			
	TRPH	See Worksheet #18	FDEP FL-PRO			
Soil	TRPH Speciation	See Worksheet #18	TPHCWG Method	21 calendar days	Lori Palmer SunLabs Inc. – Central Laboratory 5460 Beaumont Center Blvd. Suite 520 Tampa, FL 33634 813-881-9401	NA

SAP Worksheet #31 -- Planned Project Assessments Table

(UFP-QAPP Manual Section 4.1.1)

Assessment Type	Frequency	Internal or External	Organization Performing Assessment	Person(s) Responsible for Performing Assessment (title and organizational affiliation)	Person(s) Responsible for Responding to Assessment Findings (title and organizational affiliation)	Person(s) Responsible for Identifying and Implementing CA (title and organizational affiliation)	Person(s) Responsible for Monitoring Effectiveness of CA (title and organizational affiliation)
Laboratory System Audit[1]	Every two years	External	DoD ELAP Accrediting Body	DoD ELAP Accrediting Body Auditor	Laboratory QA Manager or Laboratory Manager, Empirical and SunLabs	Laboratory QAM or Laboratory Manager, Empirical and SunLabs	Laboratory QAM or Laboratory Manager, Empirical and SunLabs

1 Empirical Laboratories is DoD ELAP accredited and Florida NELAP accredited for all analytical groups and target analytes identified for this project. A copy of Empirical's accreditation is included in Appendix C. SunLabs is Florida NELAP accredited for TRPH Speciation via the TPHCWG Method. A copy of SunLab's accreditation is included in Appendix C.

SAP Worksheet #32 -- Assessment Findings and Corrective Action Responses Table
(UFP-QAPP Manual Section 4.1.2)

Assessment Type	Nature of Deficiencies Documentation	Individual(s) Notified of Findings (name, title, organization)	Timeframe of Notification	Nature of Corrective Action Response Documentation	Individual(s) Receiving Corrective Action Response (name, title, organization)	Timeframe for Response
Laboratory System Audit	Written audit report	Rick Davis, Laboratory Manager, Empirical Randy Ward, Laboratory QAM, Empirical	Specified by DoD ELAP Accrediting Body	Letter	DoD ELAP Accrediting Body	Specified by DoD ELAP Accrediting Body

SAP Worksheet #33 -- QA Management Reports Table

(UFP QAPP Manual Section 4.2)

Type of Report	Frequency (daily, weekly monthly, quarterly, annually, etc.)	Projected Delivery Date(s)	Person(s) Responsible for Report Preparation (title and organizational affiliation)	Report Recipient(s) (title and organizational affiliation)
Data validation report	Per SDG	Within 3 weeks of receipt of laboratory data package	DVM and Staff Chemists, Tetra Tech	PM and project file, Tetra Tech
Major analysis problem identification (internal Tetra Tech memorandum)	When persistent analysis problems are detected by Tetra Tech that may impact data usability	Immediately upon detection of problem (on the same day)	CLEAN QAM, Tetra Tech	PM, CLEAN QAM, Program Manager, and project file, Tetra Tech
Project monthly progress report	Monthly for duration of project	Monthly	PM, Tetra Tech	Navy RPM, Navy; CLEAN QAM, Program Manager, and project file, Tetra Tech
Laboratory QA report	When significant plan deviations result from unanticipated circumstances	Immediately upon detection of problem (on the same day)	Laboratory PM, Empirical and SunLabs	PM and project file, Tetra Tech

SAP Worksheet #34 -- Verification (Step I) Process Table
(UFP-QAPP Manual Section 5.2.1)

Verification Input	Description	Internal / External	Responsible for Verification (name, organization)
Chain-of-Custody Forms	The Tetra Tech FOL or designee will review and sign the chain-of-custody form to verify that all samples listed are included in the shipment to the laboratory and that the sample information is accurate. The forms will be signed by the sampler and a copy will be retained for the project file, Tetra Tech PM, and Tetra Tech Data Validators. See Tetra Tech SOP SA-6.3.	Internal	Sampler and FOL, Tetra Tech
SAP Sample Tables/ Chain-of-Custody Forms	Verify that all proposed samples listed in the SAP tables have been collected.	Internal	FOL or designee, Tetra Tech
Sample Log Sheets	Verify that information recorded in the log sheets is accurate and complete.	Internal	FOL or designee, Tetra Tech
SAP/ Field Logs/ Analytical Data Packages	Ensure that all sampling SOPs were followed. Verify that deviations have been documented and MPCs have been achieved. Particular attention should be given to verify that samples were correctly identified, that sampling location coordinates are accurate, and that documentation establishes an unbroken trail of documented chain-of-custody from sample collection to report generation. Verify that the correct sampling and analytical methods/SOPs were applied. Verify that the sampling plan was implemented and carried out as written and that any deviations are documented.	Internal	PM or designee, Tetra Tech
SAP/ Analytical SOPs/ Analytical Data Packages	Ensure that all laboratory SOPs were followed. Verify that the correct analytical methods/SOPs were applied.	Internal	Laboratory QAM, Empirical
SAP/ Laboratory SOPs/ Raw Data/ Applicable Control Limits Tables	Establish that all method QC samples were analyzed and in control as listed in the analytical SOPs. If method QA is not in control, the Laboratory QAM will contact the Tetra Tech PM verbally or via e-mail for guidance prior to report preparation.	Internal	Laboratory QAM, Empirical
SAP/ Chain-of-Custody Forms Electronic Data Deliverables (EDDs)/	Check that field QC samples listed in Worksheet #20 were collected as required.	Internal	FOL or designee, Tetra Tech

Verification Input	Description	Internal / External	Responsible for Verification (name, organization)
Analytical Data Packages	Each EDD will be verified against the chain-of-custody and hard copy data package for accuracy and completeness. Laboratory analytical results will be verified and compared to the electronic analytical results for accuracy. Sample results will be evaluated for laboratory contamination and will be qualified for false positives using the laboratory method/preparation blank summaries. Positive results reported between the DL and the LOQ will be qualified as estimated. Extraneous laboratory qualifiers will be removed from the validation qualifier.	External	Data Validators, Tetra Tech
Analytical Data Packages	All analytical data packages will be verified internally for completeness by the laboratory performing the work. The Laboratory QAM will sign the case narrative for each data package.	Internal	Laboratory QAM, Empirical

SAP Worksheet #35 -- Validation (Steps IIa and IIb) Process Table

(UFP-QAPP Manual Section 5.2.2) (Figure 37 UFP-QAPP Manual) (Table 9 UFP-QAPP Manual)

Step IIa / IIb[1]	Validation Input	Description	Responsible for Validation (name, organization)
IIa	SAP/ Sample Log Sheets	Sample Coordinates - Ensure that sample locations are correct and in accordance with the SAP proposed locations. Document any discrepancies in the final report.	PM, FOL, or designee, Tetra Tech
IIa	Chain-of-Custody Forms	Custody - Ensure that the custody and integrity of the samples was maintained from collection to analysis and the custody records are complete and any deviations are recorded. Review that the samples were shipped and store at the required temperature and sample pH for chemically-preserved samples meet the requirements listed in Worksheet #19. Ensure that the analyses were performed within the holding times listed in Worksheet #19.	Project Chemist or Data Validators, Tetra Tech
IIa/IIb	SAP/ Laboratory Data Packages/ EDDs	Accuracy - Ensure that the laboratory QC samples listed in Worksheet #28 were analyzed and that the MPCs listed in Worksheet #12 were met for all field samples and QC analyses. Check that specified field QC samples were collected and analyzed and that the analytical QC criteria set up for this project were met.	Project Chemist or Data Validators, Tetra Tech
		Precision - Check the field sampling precision by calculating the RPD for field duplicate samples. Check the laboratory precision by reviewing the RPD or percent difference values from laboratory duplicate analyses; MS/MSDs; and LCS/LCSD, if available. Ensure compliance with the methods and project MPCs accuracy goals listed in Worksheet #12.	
		Representativeness - Check that the laboratory recorded the temperature at sample receipt and the pH of the chemically preserved samples to ensure sample integrity from sample collection to analysis.	
		Completeness - Review the chain-of-custody forms generated in the field to ensure that the required analytical samples have been collected, appropriate sample identifications have been used, and correct analytical methods have been applied. The Tetra Tech Data Validator will verify that elements of the data package required for validation are present, and if not, the laboratory will be contacted and the missing information will be requested. Validation will be performed as per Worksheet #36. Check that all data have been transferred correctly and completely to the final SLOQ database.	

Step IIa / IIb[1]	**Validation Input**	**Description**	**Responsible for Validation** (name, organization)
IIb	SAP/ Laboratory Data Packages/ EDDs	Sensitivity - Ensure that the project LOQs listed in Worksheet #15 were achieved.	Project Chemist or Data Validators, Tetra Tech
		PALs - Discuss the impact on reported DLs due to matrix interferences or sample dilutions performed because of the high concentration of one or more other contaminants, on the other target compounds reported as non-detected. Document this usability issue and inform the Tetra Tech PM. Review and add PALs to the laboratory EDDs. Flag samples and notify the Tetra Tech PM of samples that exceed PALs listed in Worksheet #15.	
		QA/QC - Ensure that all QC samples specified in the SAP were collected and analyzed and that the associated results were within prescribed SAP acceptance limits. Ensure that QC samples and standards prescribed in analytical SOPs were analyzed and within the prescribed control limits. If any significant QC deviations occur, the Laboratory QAM shall have contacted the Tetra Tech PM.	
		Deviations - Summarize deviations from methods, procedures, or contracts in the Data Validation Report. Determine the impact of any deviation from sampling or analytical methods and SOPs requirements and matrix interferences effect on the analytical results. Qualify data results based on method or QC deviation and explain all the data qualifications. Print a copy of the project database qualified data depicting data qualifiers and data qualifiers codes that summarize the reason for data qualifications. Determine if the data met the MPCs and determine the impact of any deviations on the technical usability of the data.	

SAP Worksheet #36 –Analytical Data Validation (Steps IIa and IIb) Summary Table

(UFP-QAPP Manual Section 5.2.2.1)

Step IIa / IIb	Matrix	Analytical Group	Validation Criteria	Data Validator (title and organizational affiliation)
IIa and IIb	Soil and Groundwater	VOCs, EDB, PAHs, TRPH, and TRPH Speciation	100% Limited data validation* will be performed using criteria for SW-846 Methods 8260B, 8011, 8270C, FL-PRO, and Florida TPHCWG Method listed in Worksheets #12, #15, #24, and #28 and the DoD QSM. If not included in the aforementioned, then the logic outlined in USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review EPA-540/R-99-008 (USEPA, October 1999) will be used to apply qualifiers to data.	Data Validation Specialist, Tetra Tech
IIa and IIb	Groundwater	Total Lead	100% Limited data validation* will be performed using criteria for SW-846 Method 6010B listed in Worksheets #12, #15, #24, and #28. If not included in the aforementioned, then the logic outlined in USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review EPA 540-R-04-004 (USEPA, October 2004) will be used to apply qualifiers to data.	Data Validation Specialist, Tetra Tech

* Limited data validation. Limits the data review to specific review parameters (Data Completeness/Data Verification, Holding Times, Calibrations, Blank Contamination, & Detection Limits) to determine gross deficiencies only. The limited data validation is best expressed as a review to preclude the possibility of false negatives and to eliminate false positives. Raw data are not evaluated and sample result verification is not conducted. A formal report, similar to a full data validation report, is prepared but the scope is more limited than a full validation report. The data packages provided by the laboratory will be expansive enough to allow future complete formal data validation, if necessary.

SAP Worksheet #37 -- Usability Assessment
(UFP-QAPP Manual Section 5.2.3)

Data Usability Assessment
The usability of the data directly affects whether project objectives can be achieved. The following characteristics will be evaluated at a minimum. The results of these evaluations will be included in the project report. The characteristics will be evaluated for multiple concentration levels if the evaluator determines that this is necessary. To the extent required by the type of data being reviewed, the assessors will consult with other technically competent individuals to render sound technical assessments of these DQI characteristics: **Completeness** o For each matrix that was scheduled to be sampled, the Tetra Tech FOL acting on behalf of the Project Team will prepare a table listing planned samples/analyses to collected samples/analyses. If deviations from the scheduled sample collection or analyses are identified the Tetra Tech PM will determine whether the deviations compromise the ability to meet project objectives. If they do, the Tetra Tech PM will consult with the Navy RPM and other Project Team members, as necessary (determined by the Navy RPM), to develop appropriate corrective actions. **Precision** o The Tetra Tech Project Chemist acting on behalf of the Project Team will determine whether precision goals for field duplicates and laboratory duplicates were met. This will be accomplished by comparing duplicate results to precision goals identified in Worksheets #12 and #28. This will also include a comparison of field and laboratory precision with the expectation that field duplicate results will be no less precise than laboratory duplicate results. If the goals are not met, or data have been flagged as estimated (J qualifier), limitations on the use of the data will be described in the project report. **Accuracy** o The Tetra Tech Project Chemist acting on behalf of the project team will determine whether the accuracy/bias goals were met for project data. This will be accomplished by comparing percent recoveries of LCS, LCSD, MS, MSD, and surrogate compounds to accuracy goals identified in Worksheet #28. This assessment will include an evaluation of field and laboratory contamination; instrument calibration variability; and analyte recoveries for surrogates, matrix spike, and laboratory control samples. If the goals are not met, limitations on the use of the data will be described in the project report. Bias of the qualified results and a description of the impact of identified non-compliances on a specific data package or on the overall project data will be described in the project report. **Representativeness** o A project scientist identified by the Tetra Tech PM and acting on behalf of the project team will determine whether the data are adequately representative of intended populations, both spatially and temporally. This will be accomplished by verifying that samples were collected and processed for analysis in accordance with the SAP, by reviewing spatial and temporal data variations, and by comparing these characteristics to expectations. The usability report will describe the representativeness of the data for each matrix and analytical fraction. This will not require quantitative comparisons unless professional judgment of the project scientist indicates that a quantitative analysis is required. **Comparability** o The Tetra Tech Project Chemist acting on behalf of the Project Team will determine whether the data generated under this project are sufficiently comparable to historical site data generated by different methods and for samples collected using different procedures and under different site

conditions. This will be accomplished by comparing overall precision and bias among data sets for each matrix and analytical fraction. This will not require quantitative comparisons unless professional judgment of the Tetra Tech Project Chemist indicates that such quantitative analysis is required.

Sensitivity

- The Tetra Tech Project Chemist acting on behalf of the Project Team will determine whether project sensitivity goals listed in Worksheet #15 are achieved. The overall sensitivity and quantitation limits from multiple data sets for each matrix and analysis will be compared. If sensitivity goals are not achieved, the limitations on the data will be described. The Tetra Tech Project Chemist will enlist the help of the Tetra Tech Risk Assessor to evaluate deviations from planned sensitivity goals.

Project Assumptions and Data Outliers

- The Tetra Tech PM and designated team members will evaluate whether project assumptions are valid. This will typically be a qualitative evaluation but may be supported by quantitative evaluations. The type of evaluation depends on the assumption being tested. Quantitative assumptions include assumptions related to data distributions (e.g., Normal versus log-normal) and estimates of data variability. Statistical tests for outliers will be conducted using standard statistical techniques appropriate for this task. Potential outliers will be removed if a review of the associated indicates that the results have an assignable cause the renders them inconsistent with the rest of the data. During this evaluation, the team will consider whether outliers could be indications of unanticipated site conditions. Consideration will be given to whether outliers represent an unanticipated site condition.

Describe the evaluative procedures used to assess overall measurement error associated with the project:

After completion of the data validation, the data and data quality will be reviewed to determine whether sufficient data of acceptable quality are available for decision making. In addition to the evaluations described above, a series of inspections and statistical analyses will be performed to estimate these characteristics. The statistical evaluations will include simple summary statistics for target analytes, such as maximum concentration, minimum concentration, number of samples exhibiting non-detected results, number of samples exhibiting positive results, and the proportion of samples with detected and non-detected results. The project team members will assess whether the data collectively support the attainment of project objectives. They will consider whether any missing or rejected data have compromised the ability to make decisions or to make the decisions with the desired level of confidence. The data will be evaluated to determine whether missing or rejected data can be compensated by other data. Although rejected data will generally not be used, there may be reason to use them in a weight of evidence argument, especially when they supplement data that have not been rejected. If rejected data are used, their use will be supported by technically defensible rationales.

For statistical comparisons and mathematical manipulations, non-detected values will be represented by a concentration equal to one-half the sample-specific reporting limit. Duplicate results (original and duplicate) will not be averaged for the purpose of representing the range of concentrations. However, the average of the original and duplicate samples will be used to represent the concentration at a particular sampled location.

Identify the personnel responsible for performing the usability assessment:

The Tetra Tech PM, Project Chemist, FOL, and Project Scientist will be responsible for conducting the listed data usability assessments. The data usability assessment will be reviewed with the Navy RPM and the FDEP PM. If deficiencies affecting the attainment of project objectives are identified, the review will take place either in a face to face meeting or a teleconference depending on the extent of identified deficiencies. If no significant deficiencies are identified, the data usability assessment will simply be documented in the project report and reviewed during the normal document review cycle.

Describe the documentation that will be generated during usability assessment and how usability assessment results will be presented so that they identify trends, relationships (correlations), and anomalies: The data will be presented in tabular format, including data qualifications such as estimation (J, UJ) or rejection (R). Written documentation will support the non-compliance estimated or rejected data results. The project report will identify and describe the data usability limitations and suggest re-sampling or other corrective actions, if necessary.

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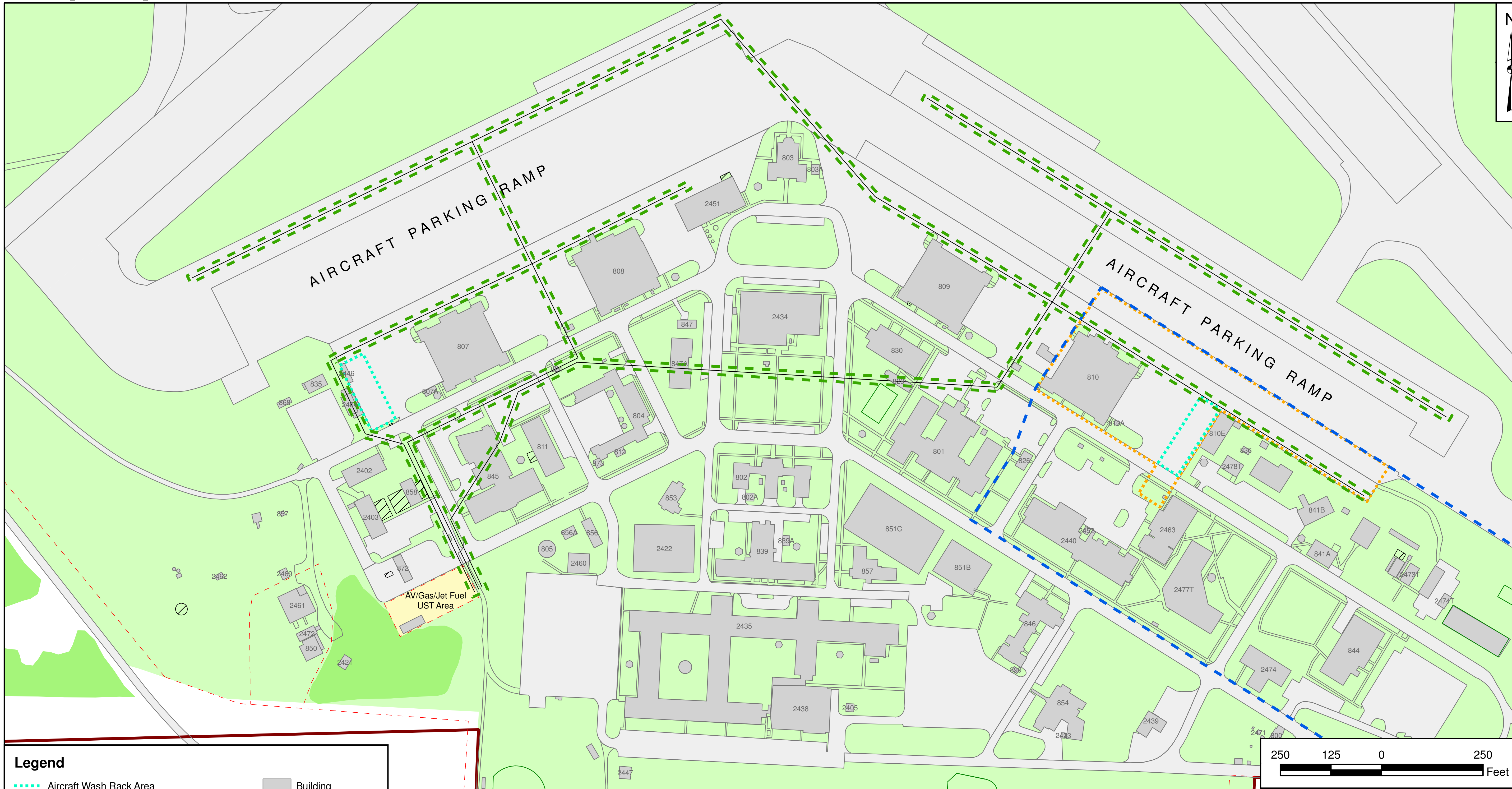
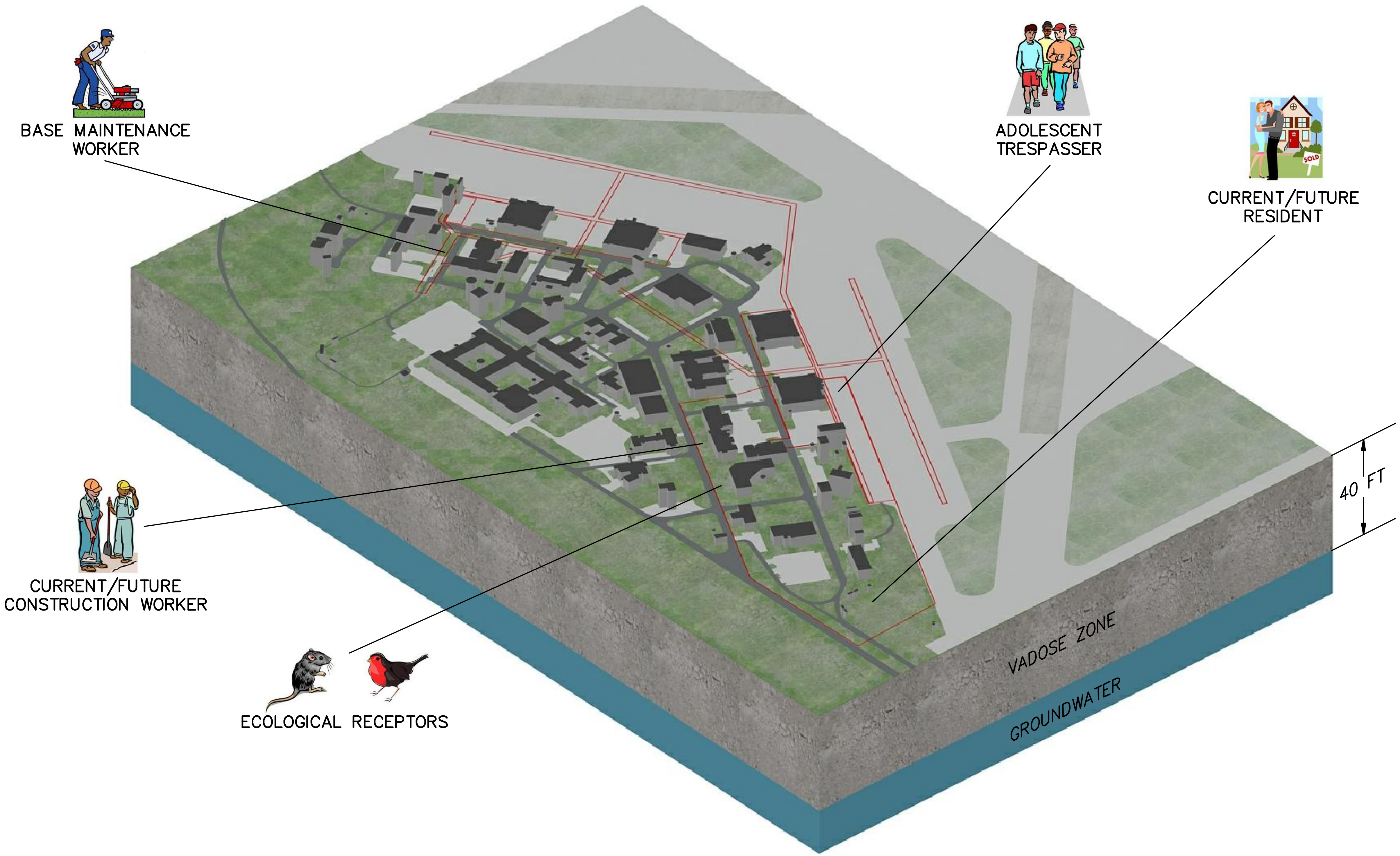
DRAWN BY K. MOORE	DATE 6/9/10	NAVFAC	BASE LOCATION MAP UFP-SAP SITE 5 OLF SAUFLEY PENSACOLA, FLORIDA	CONTRACT NUMBER —
CHECKED BY G. STIEGEL	DATE 6/9/10			APPROVED BY — DATE —
COST/SCHEDULE-AREA				APPROVED BY — DATE —
SCALE AS NOTED				FIGURE NO. FIGURE 10-1 REV 0

P:\GIS\SAUFLEY_OLF\MXD\SITE05_BASEMAP.MXD 9/17/10 KM



DRAWN BY K. MOORE	DATE 6/9/10	NAVFAC	SITE MAP UFP-SAP SITE 5 OLF SAUFLEY PENSACOLA, FLORIDA	CONTRACT NUMBER —
CHECKED BY G. POPE	DATE 9/17/10			APPROVED BY — DATE —
COST/SCHEDULE-AREA				APPROVED BY — DATE —
SCALE AS NOTED				FIGURE NO. FIGURE 10-2 — REV 0



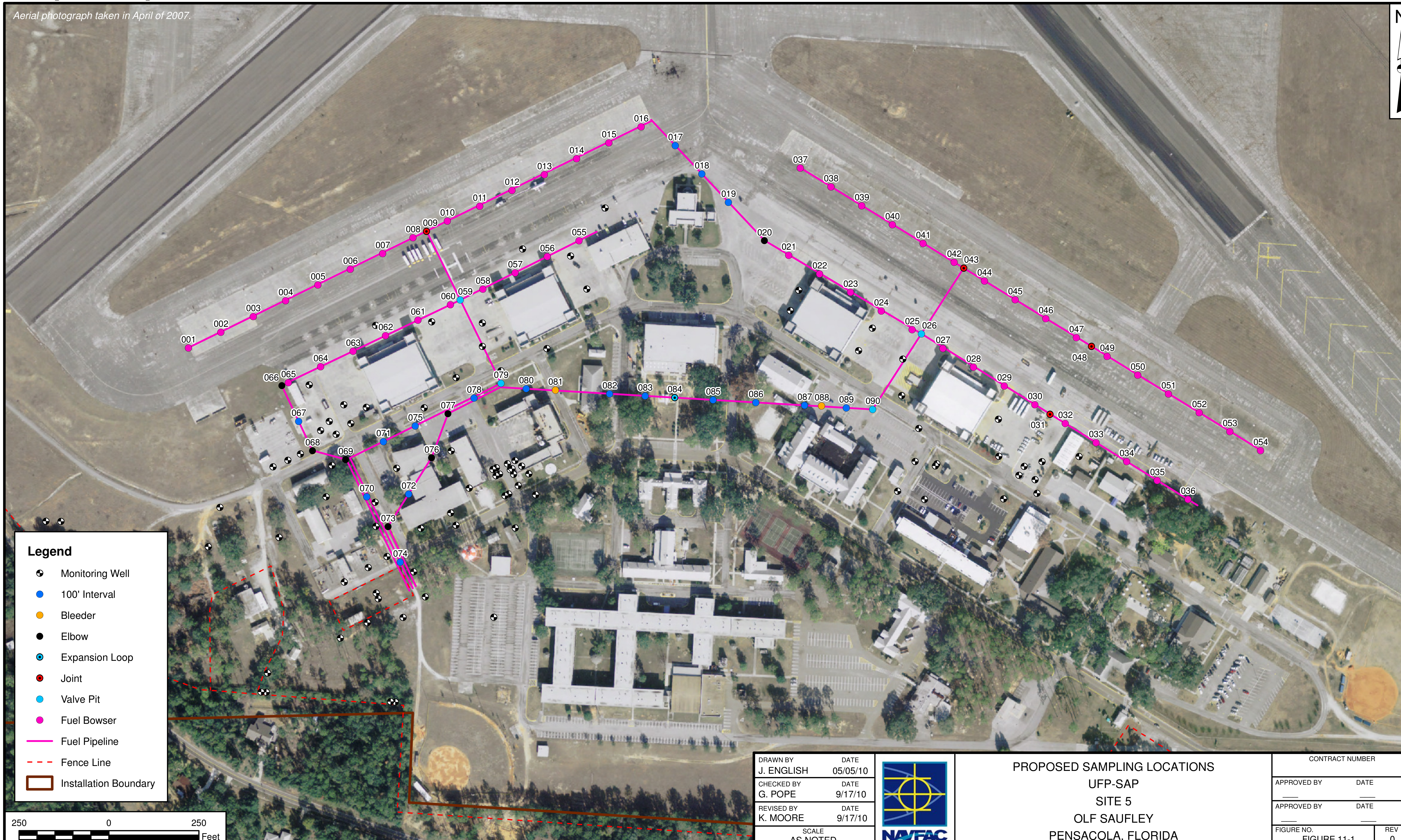
LEGEND

------ SITE 5 BOUNDARY

DRAWN BY CK / DATE 4/14/10	NAVFAC	CONCEPTUAL SITE MODEL UFP-SAP SITE 5 OLF SAUFLEY PENSACOLA, FLORIDA	CONTRACT NO. 1507	
CHECKED BY / DATE			OWNER NO.	
REVISED BY / DATE			APPROVED BY / DATE	
SCALE NOT TO SCALE			DRAWING NO. FIGURE 10-3	REV. 0

R:\2748 – OLF Saufley\CSM\2748CSM01.dwg PIT CARLY.KRAMER 6/15/2010 2:24:31 PM

P:\GIS\SAUFLEY_NOLF\MAPDOCS\MXD\FUEL_LINES.MXD 9/17/10 KM



DRAWN BY	DATE		PROPOSED SAMPLING LOCATIONS	CONTRACT NUMBER
J. ENGLISH	05/05/10	NAVFAC	UFP-SAP	
CHECKED BY	DATE		SITE 5	APPROVED BY / DATE
G. POPE	9/17/10		OLF SAUFLEY	
REVISED BY	DATE		PENSACOLA, FLORIDA	APPROVED BY / DATE
K. MOORE	9/17/10			
SCALE				FIGURE NO. / REV
AS NOTED				FIGURE 11-1 / 0

APPENDIX A

ALTERNATIVE SAMPLING PLAN

Florida Department of
Environmental Protection
Bob Martinez Center
2600 Blair Stone Road
Tallahassee, Florida 32399-2400

Charlie Crist
Governor

Jeff Kottkamp
Lt. Governor

Michael W. Sole
Secretary

June 23, 2010

Certified Mail, Return Receipt Requested
Number 7008 1830 0000 7025 8975

Ms. Sarah Reed
Naval Facilities Engineering Command Southeast
P.O. Box 30 Building 903
NAS Jacksonville, Florida 32212

Re: AP-1021
Alternative Procedures & Requirements
Outlying Landing Field Saufley Field
Pensacola, Florida
DEP Facility Number: 178628753

Dear Ms. Reed:

The Bureau of Petroleum Storage Systems has concluded its review of the Alternate Procedure request received May 25, 2010 that was submitted by Tetra Tech NUS, Inc., for the above referenced facility and enclosed as Exhibit A. The request is for relief from the requirements of Rule 62-762.801(4)(c), Florida Administrative Code (F.A.C.), (2004) for the approval of an alternative closure assessment sampling plan in place of the sampling required in the Storage Tank System Closure Assessment Requirements guidance document for closing a single-walled hydrant piping system, bowsers, valve pits and its associated appurtenances that was operated at the above referenced facility.

Pursuant to Rule 62-762.851(1), F.A.C., the Department approves the Alternate Procedure request to allow an alternative closure assessment sampling plan in place of the sampling required in the Storage Tank System Closure Assessment Requirements guidance. The closure assessment is required for any portion of the hydrant piping system that was not properly closed according to industry standards before March 12, 1991 but is acceptable for portions of the hydrant piping system that were properly closed. The closure assessment sampling and analysis shall be consistent with the plan attached as of this request. The soil samples shall be laboratory tested for the presence of the petroleum product in accordance with the general procedures in the Storage Tank System Closure Assessment Requirements guidance document and a copy of the analytical results shall be submitted to the Department's Northwest District Office. If the sample analysis exceeds the target levels contained in Department rules, an incident report in accordance with Rule 62-761.450(2), F.A.C., shall be submitted to the Department and an incident response in accordance with Chapter 62-761.820(1), F.A.C., shall be initiated.

The Department's Order shall become final unless a timely petition for an administrative hearing is filed under sections 120.569 and 120.57, F.S., within **21** days of receipt of this Order. Persons who have filed such a petition may seek to mediate the dispute and choosing mediation will not adversely affect the right to a hearing if mediation does not result in a settlement. The procedures for petitioning a hearing and pursuing mediation are set forth below.

Persons affected by this Order have the following options:

A. If you choose to accept the Department's decision regarding the Order, you do not have to do anything. This Order is final and effective as of the date on the top of the first page of this Order.

B. If you choose to challenge the decision, you may do the following:

1. File a request for an extension of time to file a petition for hearing with the Department's Agency Clerk in the Office of General Counsel within **21** days of receipt of this Order. This request should be made if you wish to meet with the Department in an attempt to resolve any disputes without first filing a petition for hearing or negotiate an agreement to mediate; or

2. File a petition for administrative hearing with the Department's Agency Clerk in the Office of General Counsel within **21** days of receipt of this Order.

3. In addition to requesting an administrative hearing, any petitioner may elect to pursue mediation under Section 120.573, F.S., and must negotiate an agreement to mediate within **10** days after the deadline for filing a petition.

<u>How to Request an Extension of Time to File a Petition for Hearing</u>

For good cause shown, pursuant to Rule 62-110.106(4), F.A.C., the Department may grant a request for an extension of time to file a petition for hearing. Such a request must be filed (received) by the Agency Clerk in the Office of General Counsel of the Department at 3900 Commonwealth Boulevard, Mail Station 35, Tallahassee, Florida, 32399-3000, within **21** days of receipt of this Order. Petitioner, if different from the applicant, shall mail a copy of the request to the applicant at the time of filing. Failure to file a petition within this time period shall waive the right of anyone who may request an administrative hearing under Sections 120.569 and 120.57, F.S.

How to File a Petition for Administrative Hearing

A person whose substantial interests are affected by this Order may petition for an administrative proceeding (hearing) under Sections 120.569 and 120.57, F.S. The petition must contain the information set forth below and must be filed (received) by the Agency Clerk in the Office of General Counsel of the Department at 3900 Commonwealth Boulevard, MS 35, Tallahassee, Florida, 32399-3000, within **21** days of receipt of this Order. Petitioner, if different from the applicant shall mail a copy of the petition to the applicant at the time of filing. Failure to file a petition within this time period shall waive the right of anyone who may request an administrative hearing under Sections 120.569 and 120.57, F.S.

Pursuant to Subsection 120.569(2), F.S., and Rule 28-106.201, F.A.C., a petition for administrative hearing shall contain the following information.

a) The name, address, and telephone number of each petitioner; the name, address, and telephone number of the petitioner's representative, if any, the site owner's name and address, if different from the petitioner, the DEP facility number, and the name and address of the facility;
b) A statement of when and how the petitioner received notice of the Department's action or proposed action;
c) An explanation of how each petitioner's substantial interests are or will be affected by the Department's action or proposed action;
d) A statement of the disputed issues of material fact, or a statement that there are no disputed facts;
e) A concise statement of the ultimate facts alleged, including a statement of the specific facts the petitioner contends warrant reversal or modification of the Department's action or proposed action;
f) A statement of the specific rules or statutes the petitioner contends requires reversal or modification of the Department's action or proposed action; and
g) A statement of the relief sought by the petitioner, stating precisely the action petitioner wishes the Department to take with respect to the Department's action or proposed action.

How to Pursue Mediation

In addition to requesting an administrative hearing, any petitioner may elect to pursue mediation. The election may be accomplished by filing with the Department a mediation agreement with all parties to the proceeding (i.e., the applicant, the Department, and any person who has filed a timely and sufficient petition for hearing). The agreement must contain all the information required by Rule 28-106.404, F.A.C. The agreement, signed by all parties, must be received by the Agency Clerk in the Office of General Counsel of the Department at 3900 Commonwealth Boulevard, Mail Station 35, Tallahassee, Florida, 32399-3000 within **10** days after the deadline for filing a petition, as set forth above. Choosing mediation will not adversely affect the right to a hearing if mediation does not result in a settlement.

Pursuant to Rule 28-106.404, F.A.C., an agreement to mediate must include the following.

(i) The name, address, and telephone number of the persons who may attend the mediation, (also the DEP facility number, the name and address of the facility if applicable);
(ii) The name, address, and telephone number of the mediator agreed to by the parties;
(iii) How the costs and fees associated with the mediation will be allocated (the Department will not pay any of the costs of mediation);
(iv) The agreement of the parties regarding the confidentiality of discussions and documents introduced during mediation to the extent authorized by law;
(v) The date, time, and place of the first mediation session;
(vi) The name of the party's representative who shall have authority to settle or recommend settlement; and
(vii) The signature of the parties.

As provided in Section 120.573, F.S., the timely agreement of all parties to mediate will toll the time limitations imposed by Sections 120.569 and 120.57, F.S., for holding an administrative hearing and issuing a final order. Unless otherwise agreed by the parties, the mediation must be concluded within sixty days of the execution of the agreement. If mediation results in settlement of the administrative dispute, the Department must enter a final order incorporating the agreement of the parties. Persons seeking to protect their substantial interests that would be affected by such a modified final decision must file their petitions within **21** days of receipt of this notice, or they shall be deemed to have waived their right to a proceeding under Sections 120.569 and 120.57, F.S. If mediation terminates without settlement of the dispute, the Department shall notify all parties in writing that the administrative hearing processes under Sections 120.569 and 120.57, F.S., are resumed.

This Order is final and effective as of the date on the top of the first page of this Order. Timely filing a petition for administrative hearing postpones the date this Order takes effect until the Department issues either a final order pursuant to an administrative hearing or mediation settlement.

Judicial Review

Any party to this Order has the right to seek judicial review of it under Section 120.68, F.S., by filing a notice of appeal under Rule 9.110 of the Florida Rules of Appellate Procedure with the Agency Clerk of the Department in the Office of General Counsel, Mail Station 35, 3900 Commonwealth Boulevard, Tallahassee, Florida 32399-3000, and by filing a copy of the notice of appeal accompanied by the applicable filing fees with the appropriate district court of appeal. The notice of appeal must be filed within thirty days after this order is filed with the clerk of the Department (see below).

Questions

Any questions regarding the Department's review of your alternate procedure should be directed to John P. Svec at (850)245-8845. Questions regarding legal issues should be referred to Rebecca Robinette, Office of General Counsel, at (850)245-2242. Contact with any of the above does not constitute a petition for administrative hearing, a request for a time extension to file a petition for hearing or an agreement to mediate.

Sincerely,

Michael E. Ashey, Chief
Bureau of Petroleum Storage Systems

MEA/jps

Enclosed: Exhibit A Petition for Alternate Procedure Request

ec: John B. Hollingshead - Escambia Co. Health Dept. – John_Hollingshead@doh.state.fl.us
Charles Harp - FDEP Northwest District Office – Charles.Harp@dep.state.fl.us
David Grabka – FDEP Federal Programs Section – David.Grabka@dep.state.fl.us
William A. Wright, Jr. – Tetra Tech NUS, Inc. - William.Wright@tetratech.com

FILING AND ACKNOWLEDGMENT
FILED, on this date, pursuant to §120.52 Florida Statutes, with the designated Department Clerk, receipt of which is hereby acknowledged.

Cheryl Stafford — 6/23/10
Clerk (or Deputy Clerk) — Date

Florida Department of Environmental Protection
Twin Towers Office Bldg.•2600 Blair Stone Road•Tallahassee, Florida 32399-2400

DEP Form # 62-761.900(4)
Form Title: Alternative Requirement or Procedure Form
Effective Date: July 13, 1998

Alternative Requirement or Procedure Form

AP# 1021

Please print or type, fill out completely, and attach additional sheets for multiple facilities.

Section 1

Facility ID No.: 8628753 County: Escambia

Facility Name: Outlying Landing Field Saufley Field

Facility Location: Pensacola, Florida

Section 2

Applicant's Name: Sarah Reed, Naval Facilities Engineering Command Southeast

Address: P.O. BOX 30 Building 903, NAS Jacksonville, FL 32212

Applicant's Telephone Number (904) 542-6290

Section 3

Rule citation within Chapter 62-761, F.A.C. that an Alternative Procedure is being requested for: 62-761.800(3)(c) (Closure Requirements)

Difference between the Chapter 62-761, F.A.C. requirement and the Alternative Procedure Request: DEP's "Storage Tank Closure Assessment Requiements" for hydrant lines require that soil samples be collected every 50 feet of linear piping for field screening. The Navy is proposing to collect a soil sample for every 100 feet of linear piping.

Please write a brief description of the proposed Alternative Procedure. (If you need additional space, please attach a separate sheet):

See attached sampling plan summary.

Section 4

Please provide a brief demonstration of how the proposed Alternative Procedure provides a substantially equivalent degree of protection for the lands, surface waters, or groundwaters of the State versus established requirements. (If you need additional space, please attach a separate sheet.)

FDEP Closure assessment requirements state that soil screening samples must be collected every 50 feet and laboratory samples must be collected for every 200 linear feet of piping. The Navy will collect laboratory samples every 100 feet to offset the extended screening interval (50 ft to 100 feet) and provide more protection.

Section 5

Sarah Reed | Sarah M Reed | 5/21/10

Applicant's Name **(Prin'** | **licant's Signature** | **Date**

EXHIBIT A

PROPOSED SAMPLING PLAN
SITE 5 – OUTLYING LANDING FIELD SAUFLEY
NAVAL AIR STATION – PENSACOLA

Site 5-Outlying Landing Field (OLF) Saufley Field (Saufley) Naval Air Station (NAS) – Pensacola consists of AVGAS fuel distribution lines (hydrant lines) that connected an underground storage tank (UST) farm to 55 refueling pits (bowsers) located along the concrete aircraft parking ramp (tarmac) on the flight line. The AVGAS fuel distribution lines were used from 1942 until 1977 when Saufley Field was decommissioned and active air operations ceased.

E.C. Jordan is believed to have performed a closure action on the bowsers in 1996. However, no closure documentation has been located. A review of the closure design drawings in April 2010 revealed that the fuel lines and bowsers located within the concrete tarmac area had once been located in concrete trenches and pits covered with steel plating. Per the closure design drawings, all bowser pits and fuel line trenches were filled with compacted soil and covered with 6-inches of concrete to grade level. In addition, several documents were located confirming E.C. Jordan's involvement in removing the bowsers at Site 5, including:

- Original tank registration correspondence registering the tanks with Florida Department of Environmental Regulation (FDER) in April 1986, including the 22 avgas tanks in bowsers (tanks 876 thru 897).
- May 1987 letter in which Navy informed FDER that the Navy had removed 35 tanks at Saufley, including tanks in bowsers (this indicates that the tanks were removed between April 1986 and May 1987).
- February 1988 letter from Navy to FDER informing FDER that E.C. Jordan was preparing tank management plans.
- June 1993 FDER Inspection Form showing the status of tanks 876 thru 897 to be "B" which is the designation tanks that have been removed.

Design drawings prepared in the 1940's detail the gasoline distribution system at Site 5 and include details of the piping, bowsers, and tank farm. From these drawings, the locations of valve pits, joints, tees, bleeders, and an expansion loop could be identified. Drawings prepared in 1985 (NAVFAC DWG #s 5134920 thru 5134925) for the pipeline capping and tank closure at Saufley Field were also located. The design drawings were prepared by the Navy Public Works Center design section and show following:

- The six 25,000 gallon fuel tanks (JP-4) and the one 15,000 gallon fuel tank (JP-4) were removed and the existing fuel lines were capped and abandoned in place at the fuel farm.
- The 500 gallon lube oil tanks located under bowsers were removed.
- The pipes were capped at the gasoline pipeline valve pits.
- Trenches and service pits were filled and paved with concrete
- As-built revision dated June 1, 1987 indicates that the contractor performed work per enclosed design drawings.

Tetra Tech has been contracted by the Navy to perform an investigation to identify and delineate areas of contamination, if present, at Site 5. The results of this investigation will be used to support closure of the pipeline under Chapter 62-761 (Underground Storage Systems), Florida Administrative Code (F.A.C.). Based on the historical information described above, and the length of pipeline requiring investigation, the following alternate closure sampling plan is proposed for approval by Florida Department of Environmental Protection:

- 1 soil sample at each bowser (55 total samples)
- 1 soil sample at each valve pit (4 total samples)
- 1 soil sample at each change in pipeline direction not associated with a valve pit (7 total samples)
- 1 soil sample at each joint between pipes of different diameter (4 total samples).
- 1 sample at the expansion loop
- 1 sample at each bleeder (2 total samples)
- 1 sample for every 100 feet (approximately) of linear buried pipe; for areas that were not enclosed in a service trench (18 total samples)
- 1 groundwater sample at every location where soil OVA measurements exceed 10 ppm and the sample depth is within 20 feet of the water table. No groundwater sample will be collected if the distance between the water table and a soil OVA exceedance (10 ppm) is greater than 20 feet.

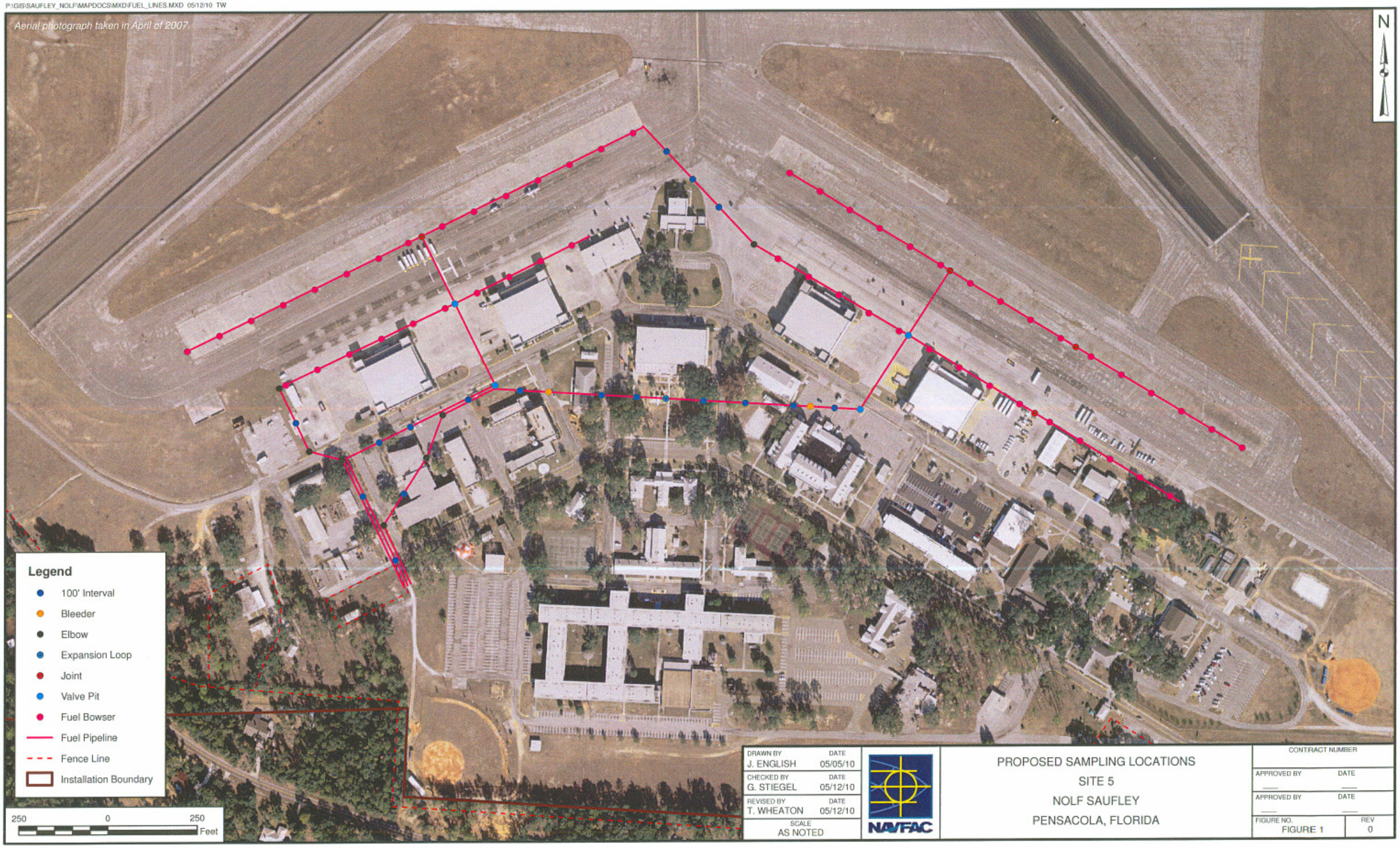
The locations of proposed soil samples are shown on the attached Figure 1.

Soil borings will be advanced no more 3 feet from the landmarks (i.e. bowsers, bleeders, valve pits, direction change, joint, and expansion loop) identified for sampling above. Soil samples for OVA-FID analysis will be collected at 2-foot vertical intervals beginning 2 foot below the estimated base of the landmark. After five samples have been collected at 2-foot intervals, additional samples will be collected at 5-foot intervals until two consecutive clean intervals are sampled or the water table is encountered.

A mobile laboratory will be on site to screen samples further using a UVF 3100 analyzer. Samples producing high, medium and low organic vapor responses will be submitted to a fixed-base laboratory for analysis of the parameters listed in Table B of Chapter 62-770, F.A.C. Laboratory analytical results will be used to confirm OVA and UVF screening data and to estimate magnitude of contamination in areas where the highest concentrations of petroleum hydrocarbons are suspected. If soil contamination is encountered in the vadose zone during Phase I, the magnitude and extent of such contamination will have been determined through UFV mobile lab screening and confirmatory laboratory analysis in the impacted areas.

Groundwater grab samples will be collected from the uppermost occurrence of groundwater (estimated to be 45 to 50 feet bls) at the boring locations. These samples collected from the screen point samplers will be analyzed for petroleum related compounds specified in Table B Chapter 62-770. If constituents of concern (COCs) are identified in these groundwater samples, a network of permanent monitoring wells will be installed to evaluate magnitude and extent of groundwater contamination.

P:\GIS\SAUFLEY_NOLF\MAPDOCS\MXD\FUEL_LINES.MXD 05/12/10 TW



APPENDIX B

FIELD STANDARD OPERATING PROCEDURES AND FIELD FORMS

Appendix B Summary

Reference #	Title, Revision
CT-04	Sample Nomenclature (Revision 2, 3/09/09)
CT-05	Database Records and Quality Assurance (Revision 2, 01/29/01)
GH-1.5	Borehole and Sample Logging (Revision 1, June, 1999)
GH-2.5	Groundwater Contour Maps and Flow Determinations (Revision 1, June, 1999)
GH-2.8	Groundwater Monitoring Well Installation (Revision 3, September, 2003)
SA-2.5	Direct Push Technology (Geoprobe®/Hydropunch™) (Revision3, 03/09/09)
HS-1.0	Utility Locating and Excavation Clearance (Revision 2, December, 2003)
SA-2.4	Soil Gas Sampling (Revision 2, September, 2003)
SA-6.1	Non-Radiological Sample Handling, Revision 3, February 2004
SA-6.3	Field Documentation Revision 3, 03/09/09)
SA-7.1	Decontamination of Field Equipment (Revision 6, 01/28/09)
SOP-05	Global Positioning System (Revision 1, July, 2010)
FC-1000	Cleaning/Decontamination Procedures, December 2008
FD-4000	Documentation for Calibration of Field-Testing Instruments and Field Analysis, December 2008
FQ-1310	Frequency, December 2008
FS-1000	General Sampling, December 2008
FS-2000	General Water Sampling, December 2008
FS-2200	Groundwater Sampling, December 2008
FS-3000	Soil Sampling, December 2008
FT-1000	General Field Testing and Measurement, December 2008
FT-1100	Field Measurement of Hydrogen Ion Activity (pH), December 2008
FT-1200	Field Measurement of Specific Conductance (Conductivity), December 2008
FT-1400	Field Measurement of Temperature, December 2008
FT-1500	Field Measurement of Dissolved Oxygen (DO), December 2008
FT-1600	Field Measurement of Turbidity, December 2008

Field Log Sheets

Boring Log

Daily Activities Checklist

Equipment Calibration Log

Groundwater Level Measurement Sheet

Chain-of-Custody Record

Monitoring Well Development Record

Overburden - Monitoring Well Sheet - Flush Mount

Field Project Pre-mobilization Checklist

QA Sample Log Sheet

TETRA TECH NUS, INC.	STANDARD OPERATING PROCEDURES	Number CT-04	Page 1 of 7
		Effective Date 03/09/09	Revision 2
		Applicability Tetra Tech NUS, Inc.	
		Prepared Earth Sciences Department	
Subject SAMPLE NOMENCLATURE		Approved Tom Johnston T.E. Johnston	

TABLE OF CONTENTS

1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to specify a consistent sample nomenclature system that will facilitate subsequent data management in a cost-effective manner. The sample nomenclature system has been devised such that the following objectives can be attained:

- Sorting of data by matrix
- Sorting of data by depth
- Maintenance of consistency (field, laboratory, and database sample numbers)
- Accommodation of all project-specific requirements
- Accommodation of laboratory sample number length constraints (maximum of 20 characters)

2.0 SCOPE

The methods described in this SOP shall be used consistently for all projects requiring electronic data. Other contract- or project-specific sample nomenclature requirements may also be applicable.

3.0 GLOSSARY

None.

4.0 RESPONSIBILITIES AND PERSONNEL QUALIFICATIONS

Program Manager - It shall be the responsibility of the Project Manager (or designee) to inform contract-specific Project Managers (PMs) of the existence and requirements of this SOP.

Project Manager - It shall be the responsibility of the PM to determine the applicability of this SOP based on: (1) program-specific requirements and (2) project size and objectives. It shall be the responsibility of the PM (or designee) to ensure that sample nomenclature requirements are thoroughly specified in the relevant project planning document (e.g., sampling and analysis plan) and are consistent with this SOP if relevant. It shall be the responsibility of the PM to ensure that the FOL is familiar with the sample nomenclature system.

Field Operations Leader (FOL) - It shall be the responsibility of the FOL to ensure that all field technicians or sampling personnel are thoroughly familiar with this SOP and the project-specific sample nomenclature system. It shall be the responsibility of the FOL to ensure that the sample nomenclature system is used during all project-specific sampling efforts.

General personnel qualifications for sample nomenclature activities in the field include the following:

- Occupational Safety and Health Administration (OSHA) 40-hour and applicable refresher training.
- Capability of performing field work under the expected physical and environmental (i.e., weather) conditions.
- Familiarity with appropriate procedures for field documentation, handling, packaging, and shipping.

5.0 PROCEDURES

5.1 INTRODUCTION

The sample identification (ID) system can consist of as few as eight but not more than 20 distinct alpha-numeric characters. The sample ID will be provided to the laboratory on the sample labels and chain-of-custody forms. The basic sample ID provided to the laboratory has three segments and shall be as follows, where "A" indicates "alpha," and "N" indicates "numeric":

A or N **3 or 4 Characters**	**AAA** **2 or 3 Characters**	**A or N** **3 to 6 Characters**
Site Identifier	Sample Type	Sample Location

Additional segments may be added as needed. For example:

(1) Soil and sediment sample ID

A or N **3 or 4 Characters**	**AAA** **2 or 3 Characters**	**A or N** **3 to 6 Characters**	**NNNN** **4 Characters**
Site identifier	Sample type	Sample location	Sample depth

(2) Aqueous (groundwater or surface water) sample ID

A or N **3 or 4 Characters**	**AAA** **2 or 3 Characters**	**A or N** **3 to 6 Characters**	**NN** **2 Characters**	**-A** **1 Character**
Site identifier	Sample type	Sample location	Round number	Filtered sample only

(3) Biota sample ID

A or N **3 or 4 Characters**	**AAA** **2 or 3 Characters**	**A or N** **3 to 6 Characters**	**AA** **2 Characters**	**NNN** **3 Characters**
Site identifier	Sample type	Sample location	Species identifier	Sample group number

5.2 SAMPLE IDENTIFICATION FIELD REQUIREMENTS

The various fields in the sample ID include but are not limited to the following:

- Site identifier
- Sample type
- Sample location
- Sample depth
- Sampling round number
- Filtered
- Species identifier
- Sample group number

The site identifier must be a three- or four-character field (numeric characters, alpha characters, or a mixture of alpha and numeric characters may be used). A site number is necessary because many facilities/sites have multiple individual sites, Solid Waste Management Units (SWMUs), Operable Units (OUs), etc. Several examples are presented in Section 5.3 of this SOP.

The sample type must be a two- or three-character alpha field. Suggested codes are provided in Section 5.3 of this SOP.

The sample location must be at least a three-character field but may have up to six characters (alpha, numeric, or a mixture). The six characters may be useful in identifying a monitoring well to be sampled or describing a grid location.

The sample depth field is used to note the depth below ground surface (bgs) at which a soil or sediment sample is collected. The first two numbers of the four-number code specify the top interval, and the third and fourth specify the bottom interval in feet bgs of the sample. If the sample depth is equal to or greater than 100, then only the top interval would be represented and the sampling depth would be truncated to three characters. The depths will be noted in whole numbers only; further detail, if needed, will be recorded on the sample log sheet or boring log, in the logbook, etc.

A two-digit round number will be used to track the number of aqueous samples collected from a particular aqueous sample location. The first sample collected from a location will be assigned the round identifier 01, the second 02, etc. This applies to both existing and proposed monitoring wells and surface water locations.

Aqueous samples that are field filtered (dissolved analysis) will be identified with an "-F" in the last field segment. No entry in this segment signifies an unfiltered (total) sample.

The species identifier must be a two-character alpha field. Several suggested codes are provided in Section 5.3 of this SOP.

The three-digit sample group number will be used to track the number of biota sample groups (a particular group size may be determined by sample technique, media type, the number of individual caught, weight issues, time, etc.) by species and location. The first sample group of a particular species collected from a given location will be assigned the sample group number 001, and the second sample group of the same species collected from the same location will be assigned the sample group number 002.

5.3 EXAMPLE SAMPLE FIELD DESIGNATIONS

Examples of each of the fields are as follows:

Site identifier - Examples of site numbers/designations are as follows:

A01 - Area of Concern (AOC) 1
125 - SWMU 125
000 - Base- or facility-wide sample (e.g., upgradient well)
BBG - Base background

The examples cited are only suggestions. Each PM (or designee) must designate appropriate (and consistent) site designations for their individual project.

Sample type - Examples of sample types are as follows:

AH - Ash Sample

AS	-	Air Sample
BM	-	Building Material Sample
BSB	-	Biota Sample Full Body
BSF	-	Biota Sample Fillet
CP	-	Composite Sample
CS	-	Chip Sample
DS	-	Drum Sample
DU	-	Dust Sample
FP	-	Free Product
IDW	-	Investigation-Derived Waste Sample
LT	-	Leachate Sample
MW	-	Monitoring Well Groundwater Sample
OF	-	Outfall Sample
RW	-	Residential Well Sample
SB	-	Soil Boring Sample
SD	-	Sediment Sample
SC	-	Scrape Sample
SG	-	Soil Gas Sample
SL	-	Sludge Sample
SP	-	Seep Sample
SS	-	Surface Soil Sample
ST	-	Storm Sewer Water Sample
SW	-	Surface Water Sample
TP	-	Test Pit Sample
TW	-	Temporary Well Sample
WC	-	Well Construction Material Sample
WP	-	Wipe Sample
WS	-	Waste/Solid Sample
WW	-	Wastewater Sample

<u>Sample location</u> - Examples of the location field are as follows:

001	-	Monitoring well 1
N32E92	-	Grid location 32 North and 92 East
D096	-	Investigation-derived waste drum number 96

<u>Species identifier</u> - Examples of species identifier are as follows:

BC	-	Blue Crab
GB	-	Blue Gill
CO	-	Corn
SB	-	Soybean

5.4 EXAMPLES OF SAMPLE NOMENCLATURE

The first round monitoring well groundwater sample collected from existing monitoring well 001 at SWMU 16 for a filtered sample would be designated as 016MW00101-F.

The second round monitoring well groundwater sample collected from existing monitoring well C20P2 at Site 23 for an unfiltered sample would be designated as 023MWC20P202.

The second surface water sample collected from point 01 at SWMU 130 for an unfiltered sample would be designated as 130SW00102.

A surface soil sample collected from grid location 32 North and 92 East at Site 32 at the 0- to 2-foot interval would be designated as 032SSN32E920002.

A subsurface soil sample from soil boring 03 at SWMU 32 at an interval of 4 to 5 feet bgs would be designated as 032SB0030405.

A sediment sample collected at SWMU 19 from 0 to 6 inches at location 14 would be designated as 019SD0140001. The sample data sheet would reflect the precise depth at which this sample was collected.

During biota sampling for full-body analysis, the first time a minnow trap was checked at grid location A25 of SWMU 1415, three small blue gills were captured, collected, and designated with the sample ID of 1415BSBA25BG001. The second time blue gill were collected at the same location (grid location A25 at SWMU 1415), the sample ID would be 1415BSBA25BG002.

Note: No dash (-) or spacing is used between the segments with the exception of the filtered segment. The "F" used for a filtered aqueous sample is preceded by a dash (-F).

5.5 FIELD QA/QC SAMPLE NOMENCLATURE

Field Quality Assurance (QA)/Quality Control (QC) samples are designated using a different coding system. The QC code will consist of a three- to four-segment alpha-numeric code that identifies the sample QC type, the date the sample was collected, and the number of this type of QC sample collected on that date.

AA	NNNNNN	NN	-F
QC type	Date	Sequence number (per day)	Filtered (aqueous only, if needed)

The QC types are identified as:

TB = Trip Blank
RB = Rinsate Blank (Equipment Blank)
FD = Field Duplicate
AB = Ambient Conditions Blank
WB = Source Water Blank

The sampling time recorded on the chain-of-custody form, labels, and tags for duplicate samples will be 0000 so that the samples are "blind" to the laboratory. Notes detailing the sample number, time, date, and type will be recorded on the routine sample log sheets and will document the location of the duplicate sample (sample log sheets are not provided to the laboratory). Documentation for all other QC types (TB, RB, AB, and WB) will be recorded on the QC Sample Log Sheet (see SOP SA-6.3, Field Documentation).

5.6 EXAMPLES OF FIELD QA/QC SAMPLE NOMENCLATURE

The first duplicate of the day for a filtered groundwater sample collected on June 3, 2000, would be designated as FD06030001-F.

The third duplicate of the day taken of a subsurface soil sample collected on November 17, 2003, would be designated as FD11170303.

The first trip blank associated with samples collected on October 12, 2000, would be designated as TB10120001.

The only rinsate blank collected on November 17, 2001, would be designated as RB11170101.

6.0 DEVIATIONS

Any deviation from this SOP must be addressed in detail in the site-specific planning documents.

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		Applicability Tetra Tech NUS, Inc.	
		Prepared Management Information Systems Department	
Subject DATABASE RECORDS AND QUALITY ASSURANCE		Approved D. Senovich	

TABLE OF CONTENTS

1.0 PURPOSE

The purpose of this document is to specify a consistent procedure for the quality assurance review of electronic and hard copy databases. This SOP outlines the requirements for establishment of a Database Record File, Quality Assurance review procedures, and documentation of the Quality Assurance Review Process.

2.0 SCOPE

The methods described in this Standard Operating Procedure (SOP) shall be used consistently for all projects managed by Tetra Tech NUS (TtNUS).

3.0 GLOSSARY

Chain-of-Custody Form - A Chain-of-Custody Form is a printed form that accompanies a sample or a group of samples from the time of sample collection to the laboratory. The Chain-of-Custody Form is retained with the samples during transfer of samples from one custodian to another. The Chain-of-Custody Form is a controlled document that becomes part of the permanent project file. Chain-of-Custody and field documentation requirements are addressed in SOP SA-6.1.

Electronic Database - A database provided on a compact laser disk (CD). Such electronic databases will generally be prepared using public domain software such as DBase, RBase, Oracle, Visual FoxPro, Microsoft Access, Paradox, etc.

Hardcopy Database - A printed copy of a database prepared using the software discussed under the definition of an electronic database.

Form I - A printed copy of the analytical results for each sample.

Sample Tracking Summary - A printed record of sample information including the date the samples were collected, the number of samples collected, the sample matrix, the laboratory to which the samples were shipped, the associated analytical requirements for the samples, the date the analytical data were received from the laboratory, and the date that validation of the sample data was completed.

4.0 RESPONSIBILITIES

Database Records Custodian - It shall be the responsibility of the Database Records Custodian to update and file the Sample Tracking Summaries for all active projects on a weekly basis. It shall be the responsibility of the Database Records Custodian to ensure that the most recent copies of the Sample Tracking Summaries are placed in the Database Records file. It shall be the responsibility of the Database Records Custodian to ensure that a copy of all validation deliverables is provided to the Project Manager (for placement in the project file). It shall be the responsibility of the Database Records Custodian to ensure that photocopies of all validation deliverables and historical data and reports (as applicable) are placed in the Database Records file.

Data Validation Coordinator - It shall be the responsibility of the Data Validation Coordinator (or designee) to ensure that the Sample Tracking Summaries are maintained by the Database Records Custodian. It shall be the responsibility of the Data Validation Coordinator (or designee) to ensure that photocopies of all data validation deliverables are placed in the applicable Database Records file by the Database Records Custodian.

Earth Sciences Department Manager - It shall be the responsibility of the Earth Sciences Department Manager (or equivalent) to ensure that all field personnel are familiar with the requirements of this Standard Operating Procedure (specifically Section 5.5).

FOL - It shall be the responsibility of the FOL (FOL) of each project to ensure that all field technicians or sampling personnel are thoroughly familiar with this SOP, specifically regarding provision of the Chain-of-Custody Forms to the Database Records Custodian. Other responsibilities of the FOL are described in Sections 5.4 and 5.5.

Management Information Systems (MIS) Manager - It shall be the responsibility of the MIS Manager to ensure that copies of original electronic deliverables (CDs) are placed in both the project files and the Database Records File. It shall be the responsibility of the MIS Manager (or designee) to verify the completeness of the database (presence of all samples) in both electronic and hardcopy form in the Database Records File. It shall be the responsibility of the MIS Manager to ensure that Quality Assurance Reviews are completed and are attested to by Quality Assurance Reviewers. It shall be the responsibility of the MIS Manager to ensure that records of the Quality Assurance review process are placed in the Database Records File. It shall be the responsibility of the MIS Manager to ensure that both electronic and hardcopy forms of the final database are placed in both the project and the Database Record File. It shall be the responsibility of the MIS Manager to ensure that data validation qualifiers are entered in the database.

Furthermore, it shall be the responsibility of the MIS Manager to participate in project planning at the request of the Project Manager, specifically with respect to the generation of level of effort and schedule estimates. To support the project planning effort, the MIS Manager shall provide a copy of the MIS Request From included as Attachment A to the project manager. It shall be the responsibility of the MIS Manager to generate level of effort and budget estimates at the time database support is requested if a budget does not exist at the time of the request. The MIS Request Form shall be provided to the Project Manager at the time of any such requests. It shall be the responsibility of the MIS Manager to notify the Project Manager of any anticipated level of effort overruns or schedule noncompliances as soon as such problems arise along with full justification for any deviations from the budget estimates (provided they were generated by the MIS Manager). It shall be the responsibility of the MIS Manager to document any changes to the scope of work dictated by the Project Manager, along with an estimate of the impact of the change on the level of effort and the schedule.

Program/Department Managers - It shall be the responsibility of the Department and/or Program Managers (or designees) to inform their respective department's Project Managers of the existence and requirements of this SOP.

Project Manager - It shall be the responsibility of each Project Manager to determine the applicability of this SOP based on: (1) program-specific requirements, and (2) project size and objectives. It shall be the responsibility of the Project Manager (or designee) to ensure that the FOL is familiar with the requirements regarding Chain-of-Custody Form provision to the Database Records Custodian. It shall be the responsibility of the Project Manager (or designee) to determine which, if any, historical data are relevant and to ensure that such data (including all relevant information such as originating entity, sample locations, sampling dates, etc.) are provided to the Database Records Custodian for inclusion in the Database Records File. It shall be the responsibility of the Project Manager to obtain project planning input regarding the level of effort and schedule from the MIS Manager. It shall be the responsibility of the Project Manager to complete the database checklist (Attachment A) to support the level of effort and schedule estimate and to facilitate database preparation and subroutine execution.

Risk Assessment Department Manager - It shall be the responsibility of the Risk Assessment Department Manager to monitor compliance with this Standard Operating Procedure, to modify this SOP as necessary, and to take corrective action if necessary. Monitoring of the process shall be completed on a quarterly basis.

Quality Assurance Reviewers - It shall be the responsibility of the Quality Assurance Reviewers to verify the completeness of the sample results via review of the Chain-of-Custody Forms and Sample Tracking Summaries. It shall be the responsibility of the Quality Assurance Reviewers to ensure the correctness of the database via direct comparison of the hardcopy printout of the database and the hardcopy summaries of the original analytical data (e.g., Form Is provided in data validation deliverables). Correctness includes the presence of all relevant sample information (all sample information fields), agreement of the laboratory and database analytical results, and the presence of data validation qualifiers.

Quality Manager - It shall be the responsibility of the Quality Manager to monitor compliance with this Standard Operating Procedure via routine audits.

5.0 PROCEDURES

5.1 Introduction

Verification of the accuracy and completeness of an electronic database can only be accomplished via comparison of a hardcopy of the database with hardcopy of all relevant sample information. The primary purposes of this SOP are to ensure that 1) all necessary hardcopy information is readily available to Quality Assurance Reviewers; 2) ensure that the Quality Assurance review is completed in a consistent and comprehensive manner, and; 3) ensure that documentation of the Quality Assurance review process is maintained in the project file.

5.2 File Establishment

A Database Record file shall be established for a specific project at the discretion of the Project Manager. Initiation of the filing procedure will commence upon receipt of the first set of Chain-of-Custody documents from a FOL or sampling technician. The Database Record Custodian shall establish a project-specific file for placement in the Database Record File. Each file in the Database Record File shall consist of standard components placed in the file as the project progresses. Each file shall be clearly labeled with the project number, which shall be placed on the front of the file drawer and on each and every hanging file folder relevant to the project. The following constitute the minimum components of a completed file:

- Electronic Deliverables
- Sample Tracking Forms
- Chain-of-Custody Forms
- Data Validation Letters
- Quality Assurance Records

5.3 Electronic Deliverables

The format of electronic deliverables shall be specified in the laboratory procurement specification and shall be provided by the laboratory. The integrity of all original electronic data deliverables shall be maintained. This shall be accomplished via the generation of copies of each electronic deliverable provided by the laboratory. The original electronic deliverable shall be provided to the project manager for inclusion in the project file. A copy of the original electronic deliverable shall be placed in the Database Record File. The second copy shall be maintained by the MIS Manager (or designee) to be used as a working copy.

5.4 Sample Tracking Forms

Updated versions of the sample tracking form for each relevant project shall be maintained by the Database Record Custodian. The Sample Tracking Forms shall be updated any time additional Chain-of-Custody Forms are received from a FOL or sampling technician, or at any time that data are received from a laboratory, or at any time that validation of a given data package (sample delivery group) is completed. The Data Validation Coordinator shall inform the Database Record Custodian of the receipt of any data packages from the laboratory and of completion of validation of a given data package to facilitate updating of the Sample Tracking Form. The Database Record Custodian shall place a revised copy of the Sample Tracking Form in the Database Record File anytime it has been updated. Copies of the updated Sample Tracking Form shall also be provided to the project manager to apprise the project manager of sample package receipt, completion of validation, etc.

5.5 Chain-of-Custody Forms

The Chain-of-Custody Forms for all sampling efforts will be used as the basis for (1) updating the Sample Tracking Form, and (2) confirming that all required samples and associated analyses have been completed. It shall be the responsibility of the FOL (or sample technician) to provide a photocopy of all Chain-of-Custody Forms to the Database Record Custodian immediately upon completion of a sampling effort. The Database Record Custodian shall then place the copies of the Chain-of-Custody Form(s) in the Database Record File. Upon receipt of a sample data package from an analytical laboratory, the Data Validation Coordinator shall provide a copy of the laboratory Chain-of-Custody Form to the Database Record Custodian. The Database Record Custodian shall use this copy to update the Sample Tracking Summary and shall place the copy of the laboratory-provided Chain-of-Custody Form in the Database Record File. The photocopy of the laboratory-provided Chain-of Custody Form shall be stapled to the previously filed field copy. Upon receipt of all analytical data, two copies of the Chain-of-Custody will therefore be in the file. Review of the Chain-of-Custody Forms will therefore be a simple mechanism to determine if all data have been received. Chain-of-Custody is addressed in SOP SA-6.1.

5.6 Data Validation Letters

All data validation deliverables (or raw data summaries if validation is not conducted) shall be provided for inclusion in both the Database Record File and the project file. If USEPA regional- or client-specific requirements are such that Form Is (or similar analytical results) need not be provided with the validation deliverable, copies of such results must be appended to the deliverable. It is preferable, although not essential that the validation qualifiers be hand-written directly on the data summary forms. The data validation deliverables (and attendant analytical summaries) will provide the basis for direct comparison of the database printout and the raw data and qualifiers.

5.7 Historical Data

At the direction of the Project Manager, historical data may also be included in a project-specific analytical database. In the event that historical data are germane to the project, hardcopy of the historical data must be included in the Database Record File. Historical data may be maintained in the form of final reports or as raw data. The information contained in the historical data file must be sufficient to identify its origin, its collection date, the sample location, the matrix, and any and all other pertinent information. All available analytical data, Chain-of-Custody Forms, boring logs, well construction logs, sample location maps, shall be photocopied by the Project Manager (or designee) and placed in one or more 3-ring binders. All information shall be organized chronologically by matrix. It shall be the responsibility of the Project Manager (or designee) to ensure that all inconsistencies between analytical data, Chain-of-Custody Forms, boring logs, sample log sheets, and field logbooks are identified and corrected. The Project Manager (or designee) shall decide which nomenclature is appropriate and edit, initial and date all relevant forms. Data entry may only be performed on information that has undergone the aforementioned editing process, thereby having a direct correlation between hardcopy information and what will become the electronic database.

6.0 RECORDS

Records regarding database preparation and quality assurance review include all those identified in the previous section. Upon completion of the database task, records from the file will be forwarded to the Project Manager for inclusion in the project file, or will be placed in bankers boxes (or equivalent) for storage. The final records for storage shall include the following minimum information on placards placed on both the top and end of the storage box:

Database Record File
PROJECT NUMBER: ____
SITE NAME: _________
DATE FILED: __/__/__
SUMMARY OF CONTENTS ENCLOSED
BOX _ OF _

Project- or program-specific record keeping requirements shall take precedence over the record keeping requirements of this SOP.

ATTACHMENT A

MIS REQUEST FORM

Tetra Tech NUS, Inc.

Project Name: ____ Request Date: ____
CTO: ____ Date Data Available for Production: ____
Project Manager: ____ Request in Support of: ____
Requestor: ____ Database Lead: ____
Program/Client: ____ GIS Lead: ____
State/EPA Region: ____ Statistics Lead: ____
Risk Lead: ____
Site Name(s) (Area, OU, etc.): ____
Sampling Date(s): ____
Matrix: ☐ GW ☐ SO ☐ SD ☐ SW ☐ Other: ____

Labels: ☐ Labels needed for an upcoming sampling event ____ Total # of Samples
____ Estimated Hours Additional Instructions: ____
____ Due Date
____ Complete ETS Charge No.
____ FOL

Data Entry:
☐ Chemical data needs to be entered from hardcopy ____ Estimated # of Samples
☐ Chemical data needs to be formated electronically
☐ Field analytical data needs to be entered from hardcopy
☐ Geologic data needs to be entered from hardcopy
☐ Hydrology data needs to be entered from hardcopy
____ Estimated Hours Additional Instructions: ____
____ Due Date
____ Complete ETS Charge No.

Tables:
☐ Full Data Printout
☐ Summary of Positive Hits
☐ Occurance and Distribution ☐ with criteria
☐ Sampling Analytical Summary
☐ Other: ____
____ Estimated Hours Additional Instructions: ____
____ Due Date
____ Complete ETS Charge No.

GIS:
☐ General Facility Location
☐ Site Location
☐ Potentiometric Contours/Groundwater Flow
☐ Sample Location Proposed
☐ Sample Location Existing
☐ Tag Map Single Round
☐ Tag Map Multiple Round
☐ Isoconcentrations
☐ Chart Map
☐ 3D Visualization
☐ EGIS CD
☐ Other: ____
____ Estimated Hours Additional Instructions: ____
____ Due Date
____ Complete ETS Charge No.

Statistics: ☐ Yes
____ Estimated Hours Additional Instructions: ____
____ Due Date
____ Complete ETS Charge No.

Geostatistics: ☐ Yes
____ Estimated Hours Additional Instructions: ____
____ Due Date
____ Complete ETS Charge No.

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		Applicability Tetra Tech NUS, Inc.	
		Prepared Earth Sciences Department	
Subject BOREHOLE AND SAMPLE LOGGING		Approved D. Senovich	

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FIGURES

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1.0 PURPOSE

The purpose of this document is to establish standard procedures and technical guidance on borehole and sample logging.

2.0 SCOPE

These procedures provide descriptions of the standard techniques for borehole and sample logging. These techniques shall be used for each boring logged to provide consistent descriptions of subsurface lithology. While experience is the only method to develop confidence and accuracy in the description of soil and rock, the field geologist/engineer can do a good job of classification by careful, thoughtful observation and by being consistent throughout the classification procedure.

3.0 GLOSSARY

None.

4.0 RESPONSIBILITIES

Site Geologist. Responsible for supervising all boring activities and assuring that each borehole is completely logged. If more than one rig is being used on site, the Site Geologist must make sure that each field geologist is properly trained in logging procedures. A brief review or training session may be necessary prior to the start up of the field program and/or upon completion of the first boring.

5.0 PROCEDURES

The classification of soil and rocks is one of the most important jobs of the field geologist/engineer. To maintain a consistent flow of information, it is imperative that the field geologist/engineer understand and accurately use the field classification system described in this SOP. This identification is based on visual examination and manual tests.

5.1 Materials Needed

When logging soil and rock samples, the geologist or engineer may be equipped with the following:

- Rock hammer
- Knife
- Camera
- Dilute hydrochloric acid (HCl)
- Ruler (marked in tenths and hundredths of feet)
- Hand Lens

5.2 Classification of Soils

All data shall be written directly on the boring log (Figure 1) or in a field notebook if more space is needed. Details on filling out the boring log are discussed in Section 5.5.

FIGURE 1

BORING LOG (EXAMPLE)

BORING LOG

Page ___ of ___

PROJECT NAME: ______________________ BORING NUMBER: ______________________

PROJECT NUMBER: ______________________ DATE: ______________________

DRILLING COMPANY: ______________________ GEOLOGIST: ______________________

DRILLING RIG: ______________________ DRILLER: ______________________

Sample No. and Type or RQD	Depth (Ft.) or Run No.	Blows / 6" or RQD (%)	Sample Recovery / Sample Length	Lithology Change (Depth/Ft.) or Screened Interval	MATERIAL DESCRIPTION			U S C S *	Remarks	PID/FID Reading (ppm)			
					Soil Density/ Consistency or Rock Hardness	Color	Material Classification			Sample	Sampler BZ	Borehole**	Driller BZ**

* When rock coring, enter rock brokeness.

** Include monitor reading in 6 foot intervals @ borehole. Increase reading frequency if elevated response read.

Remarks: ______________________ Drilling Area Background (ppm): ____

Converted to Well: Yes ______ No ______ Well I.D. #: ______________________

FIGURE 1 (CONTINUED)

SOIL TERMS

UNIFIED SOIL CLASSIFICATION (USCS)

COARSE-GRAINED SOILS — More Than Half of Material is LARGER Than No. 200 Sieve Size

FIELD IDENTIFICATION PROCEDURES (Excluding Particles Larger Than 3 Inches and Basing Fractions on Estimated Weights)			GROUP SYMBOL	TYPICAL NAMES
GRAVELS 50% (+) > 1/4" ø	CLEAN GRAVELS (Low % Fines)	Wide range in grain size and substantial amounts of all intermediate particle sizes.	GW	Well graded gravels, gravel-sand mixtures, little or no fines.
		Predominantly one size or a range of sizes with some intermediate sizes missing.	GP	Poorly graded gravels, gravel-sand mixtures, little or no fines.
	GRAVELS W/FINES (High % Fines)	Non-plastic fines (for identification procedures, see ML)	GM	Silty gravels, poorly graded gravel-sand-silt mixtures.
		Plastic fines (for identification procedures, see CL)	GC	Clayey gravels, poorly graded gravel-sand-clay mixtures.
SANDS 50% (+) < 1/4" ø	CLEAN SANDS (Low % Fines)	Wide range in grain size and substantial amounts of all intermediate particle sizes.	SW	Well graded sand, gravelly sands, little or no fines.
		Predominantly one size or a range of sizes with some intermediate sizes missing.	SP	Poorly graded sands, gravelly sands, little or no fines.
	SANDS W/FINES (High % Fines)	Non-plastic fines (for identification procedures, see ML)	SM	Silty sands, poorly graded sand-silt mixtures.
		Plastic fines (for identification procedures, see CL)	SC	Clayey sands, poorly graded sand-clay mixtures.

FINE-GRAINED SOILS — More Than Half of Material is SMALLER Than No. 200 Sieve Size

FIELD IDENTIFICATION PROCEDURES (Excluding Particles Larger Than 3 Inches and Basing Fractions on Estimated Weights) — Identification Procedures on Fraction Smaller than No. 40 Sieve Size	DRY STRENGTH (Crushing Characteristics)	DILATANCY (Reaction to Shaking)	TOUGHNESS (Consistency Near Plastic Limit)	GROUP SYMBOL	TYPICAL NAMES
SILTS AND CLAYS Liquid Limit < 50	None to Slight	Quick to Slow	None	ML	Inorganic silts and very fine sands, rock flour, silty or clayey fine sands with slight plasticity.
	Medium to High	None to Very Slow	Medium	CL	Inorganic clays of low to medium plasticity, gravelly clays, sandy clays, silty clays, lean clays.
	Slight to Medium	Slow	Slight	OL	Organic silts and organic silt-clays of low plasticity.
SILTS AND CLAYS Liquid Limit > 50	Slight to Medium	Slow to None	Slight to Medium	MH	Inorganic silts, micaceous or diatomaceous fine sandy or silty soils, elastic silts.
	High to Very High	None	High	CH	Inorganic clays of high plasticity, fat clays.
	Medium to High	None to Very Slow	Slight to Medium	OH	Organic clays of medium to high plasticity.
HIGHLY ORGANIC SOILS	Readily identified by color, odor, spongy feel and frequently by fibrous texture.			Pt	Peat and other organic soils

Boundary classifications: Soils possessing characteristics of two groups are designated by combining group symbols. For example, GW-GC, well graded gravel-sand mixture with clay binder.
All sieve sizes on this chart are U.S. Standard.

DENSITY OF GRANULAR SOILS

DESIGNATION	STANDARD PENETRATION RESISTANCE-BLOWS/FOOT
Very Loose	0-4
Loose	5-10
Medium Loose	11-30
Dense	31-50
Very Dense	Over 50

CONSISTENCY OF COHESIVE SOILS

CONSISTENCY	UNC. COMPRESSIVE STRENGTH (TONS/SQ.FT.)	STANDARD PENETRATION RESISTANCE-BLOWS/FOOT	FIELD IDENTIFICATION METHODS
Very Soft	Less than 0.25	0 to 2	Easily penetrated several inches by fist
Soft	0.25 to 0.50	2 to 4	Easily penetrated several inches by thumb.
Medium Stiff	0.50 to 1.0	4 to 8	Can be penetrated several inches by thumb.
Stiff	1.0 to 2.0	8 to 15	Readily indented by thumb.
Very Stiff	2.0 to 4.0	15 to 30	Readily indented by thumbnail.
Hard	More than 4.0	Over 30	Indented with difficulty by thumbnail.

ROCK TERMS

ROCK HARDNESS (FROM CORE SAMPLES)			ROCK BROKENNESS		
Descriptive Terms	Screwdriver or Knife Effects	Hammer Effects	Descriptive Terms	Abbreviation	Spacing
Soft	Easily Gouged	Crushes when pressed with hammer	Very Broken	(V. Br.)	0-2"
Medium Soft	Can be Gouged	Breaks (one blow); crumbly edges	Broken	(Br.)	2"-1'
Medium Hard	Can be scratched	Breaks (one blow); sharp edges	Blocky	(Bl.)	1'-3'
Hard	Cannot be scratched	Breaks conchoidally (several blows); sharp edges	Massive	(M.)	3'-10'

LEGEND:

SOIL SAMPLES - TYPES

S - 2" Split-Barrel Sample
ST - 3" O.D. Undisturbed Sample
O - Other Samples, Specify in Remarks

ROCK SAMPLES - TYPES

X - NX (Conventional) Core (~2-1/8" O.D.)
Q - NQ (Wireline) Core (~1-7/8" O.D.)
Z - Other Core Sizes, Specify in Remarks

WATER LEVELS

12/18 ▽ 12.6' Initial Level w/Date & Depth
12/18 ▼ 12.6' Stabilized Level w/Date & Depth

5.2.1 USCS Classification

Soils are to be classified according to the Unified Soil Classification System (USCS). This method of classification is detailed in Figure 1 (Continued).
This method of classification identifies soil types on the basis of grain size and cohesiveness.

Fine-grained soils, or fines, are smaller than the No. 200 sieve and are of two types: silt (M) and clay (C). Some classification systems define size ranges for these soil particles, but for field classification purposes, they are identified by their respective behaviors. Organic material (O) is a common component of soil but has no size range; it is recognized by its composition. The careful study of the USCS will aid in developing the competence and consistency necessary for the classification of soils.

Coarse-grained soils shall be divided into rock fragments, sand, or gravel. The terms sand and gravel not only refer to the size of the soil particles but also to their depositional history. To insure accuracy in description, the term rock fragments shall be used to indicate angular granular materials resulting from the breakup of rock. The sharp edges typically observed indicate little or no transport from their source area, and therefore the term provides additional information in reconstructing the depositional environment of the soils encountered. When the term "rock fragments" is used it shall be followed by a size designation such as "(1/4 inchΦ-1/2 inchΦ)" or "coarse-sand size" either immediately after the entry or in the remarks column. The USCS classification would not be affected by this variation in terms.

5.2.2 Color

Soil colors shall be described utilizing a single color descriptor preceded, when necessary, by a modifier to denote variations in shade or color mixtures. A soil could therefore be referred to as "gray" or "light gray" or "blue-gray." Since color can be utilized in correlating units between sampling locations, it is important for color descriptions to be consistent from one boring to another.

Colors must be described while the sample is still moist. Soil samples shall be broken or split vertically to describe colors. Samplers tend to smear the sample surface creating color variations between the sample interior and exterior.

The term "mottled" shall be used to indicate soils irregularly marked with spots of different colors. Mottling in soils usually indicates poor aeration and lack of good drainage.

Soil Color Charts shall not be used unless specified by the project manager.

5.2.3 Relative Density and Consistency

To classify the relative density and/or consistency of a soil, the geologist is to first identify the soil type. Granular soils contain predominantly sands and gravels. They are noncohesive (particles do not adhere well when compressed). Finer-grained soils (silts and clays) are cohesive (particles will adhere together when compressed).

The density of noncohesive, granular soils is classified according to standard penetration resistances obtained from split-barrel sampling performed according to the methods detailed in Standard Operating Procedures GH-1.3 and SA-1.3. Those designations are:

Designation	Standard Penetration Resistance (Blows per Foot)
Very loose	0 to 4
Loose	5 to 10
Medium dense	11 to 30
Dense	31 to 50
Very dense	Over 50

Standard penetration resistance is the number of blows required to drive a split-barrel sampler with a 2-inch outside diameter 12 inches into the material using a 140-pound hammer falling freely through 30 inches. The sampler is driven through an 18-inch sample interval, and the number of blows is recorded for each 6-inch increment. The density designation of granular soils is obtained by adding the number of blows required to penetrate the last 12 inches of each sample interval. It is important to note that if gravel or rock fragments are broken by the sampler or if rock fragments are lodged in the tip, the resulting blow count will be erroneously high, reflecting a higher density than actually exists. This shall be noted on the log and referenced to the sample number. Granular soils are given the USCS classifications GW, GP, GM, SW, SP, SM, GC, or SC (see Figure 1).

The consistency of cohesive soils is determined by performing field tests and identifying the consistency as shown in Figure 2.

Cohesive soils are given the USCS classifications ML, MH, CL, CH, OL, or OH (see Figure 1).

The consistency of cohesive soils is determined either by blow counts, a pocket penetrometer (values listed in the table as Unconfined Compressive Strength), or by hand by determining the resistance to penetration by the thumb. The pocket penetrometer and thumb determination methods are conducted on a selected sample of the soil, preferably the lowest 0.5 foot of the sample in the split-barrel sampler. The sample shall be broken in half and the thumb or penetrometer pushed into the end of the sample to determine the consistency. Do not determine consistency by attempting to penetrate a rock fragment. If the sample is decomposed rock, it is classified as a soft decomposed rock rather than a hard soil. Consistency shall not be determined solely by blow counts. One of the other methods shall be used in conjunction with it. The designations used to describe the consistency of cohesive soils are shown in Figure 2.

5.2.4 Weight Percentages

In nature, soils are comprised of particles of varying size and shape, and are combinations of the various grain types. The following terms are useful in the description of soil:

Terms of Identifying Proportion of the Component	Defining Range of Percentages by Weight
Trace	0 - 10 percent
Some	11 - 30 percent
Adjective form of the soil type (e.g., "sandy")	31 - 50 percent

FIGURE 2

CONSISTENCY FOR COHESIVE SOILS

Consistency	Standard Penetration Resistance (Blows per Foot)	Unconfined Compressive Strength (Tons/Sq. Foot by pocket penetration)	Field Identification
Very soft	0 to 2	Less than 0.25	Easily penetrated several inches by fist
Soft	2 to 4	0.25 to 0.50	Easily penetrated several inches by thumb
Medium stiff	4 to 8	0.50 to 1.0	Can be penetrated several inches by thumb with moderate effort
Stiff	8 to 15	1.0 to 2.0	Readily indented by thumb but penetrated only with great effort
Very stiff	15 to 30	2.0 to 4.0	Readily indented by thumbnail
Hard	Over 30	More than 4.0	Indented with difficulty by thumbnail

Examples:

- Silty fine sand: 50 to 69 percent fine sand, 31 to 50 percent silt.
- Medium to coarse sand, some silt: 70 to 80 percent medium to coarse sand, 11 to 30 percent silt.
- Fine sandy silt, trace clay: 50 to 68 percent silt, 31 to 49 percent fine sand, 1 to 10 percent clay.
- Clayey silt, some coarse sand: 70 to 89 percent clayey silt, 11 to 30 percent coarse sand.

5.2.5 Moisture

Moisture content is estimated in the field according to four categories: dry, moist, wet, and saturated. In dry soil, there appears to be little or no water. Saturated samples obviously have all the water they can hold. Moist and wet classifications are somewhat subjective and often are determined by the individual's judgment. A suggested parameter for this would be calling a soil wet if rolling it in the hand or on a porous surface liberates water, i.e., dirties or muddies the surface. Whatever method is adopted for describing moisture, it is important that the method used by an individual remains consistent throughout an entire drilling job.

Laboratory tests for water content shall be performed if the natural water content is important.

5.2.6 Stratification

Stratification can only be determined after the sample barrel is opened. The stratification or bedding thickness for soil and rock is depending on grain size and composition. The classification to be used for stratification description is shown in Figure 3.

5.2.7 Texture/Fabric/Bedding

The texture/fabric/bedding of the soil shall be described. Texture is described as the relative angularity of the particles: rounded, subrounded, subangular, and angular. Fabric shall be noted as to whether the particles are flat or bulky and whether there is a particular relation between particles (i.e., all the flat particles are parallel or there is some cementation). The bedding or structure shall also be noted (e.g., stratified, lensed, nonstratified, heterogeneous varved).

5.2.8 Summary of Soil Classification

In summary, soils shall be classified in a similar manner by each geologist/engineer at a project site. The hierarchy of classification is as follows:

- Density and/or consistency
- Color
- Plasticity (Optional)
- Soil types
- Moisture content
- Stratification
- Texture, fabric, bedding
- Other distinguishing features

FIGURE 3

BEDDING THICKNESS CLASSIFICATION

Thickness (metric)	Thickness (Approximate English Equivalent)	Classification
> 1.0 meter	> 3.3'	Massive
30 cm - 1 meter	1.0' - 3.3'	Thick Bedded
10 cm - 30 cm	4" - 1.0'	Medium Bedded
3 cm - 10 cm	1" - 4"	Thin Bedded
1 cm - 3 cm	2/5" - 1"	Very Thin Bedded
3 mm - 1 cm	1/8" - 2/5"	Laminated
1 mm - 3 mm	1/32" - 1/8"	Thinly Laminated
< 1 mm	<1/32"	Micro Laminated

(Weir, 1973 and Ingram, 1954)

5.3 Classification of Rocks

Rocks are grouped into three main divisions: sedimentary, igneous and metamorphic. Sedimentary rocks are by far the predominant type exposed at the earth's surface. The following basic names are applied to the types of rocks found in sedimentary sequences:

- Sandstone - Made up predominantly of granular materials ranging between 1/16 to 2 mm in diameter.
- Siltstone - Made up of granular materials less than 1/16 to 1/256 mm in diameter. Fractures irregularly. Medium thick to thick bedded.
- Claystone - Very fine-grained rock made up of clay and silt-size materials. Fractures irregularly. Very smooth to touch. Generally has irregularly spaced pitting on surface of drilled cores.
- Shale - A fissile very fine-grained rock. Fractures along bedding planes.
- Limestone - Rock made up predominantly of calcite ($CaCO_3$). Effervesces strongly upon the application of dilute hydrochloric acid.
- Coal - Rock consisting mainly of organic remains.
- Others - Numerous other sedimentary rock types are present in lesser amounts in the stratigraphic record. The local abundance of any of these rock types is dependent upon the depositional history of the area. Conglomerate, halite, gypsum, dolomite, anhydrite, lignite, etc. are some of the rock types found in lesser amounts.

In classifying a sedimentary rock the following hierarchy shall be noted:

- Rock type
- Color
- Bedding thickness
- Hardness
- Fracturing
- Weathering
- Other characteristics

5.3.1 Rock Type

As described above, there are numerous types of sedimentary rocks. In most cases, a rock will be a combination of several grain types, therefore, a modifier such as a sandy siltstone, or a silty sandstone can be used. The modifier indicates that a significant portion of the rock type is composed of the modifier. Other modifiers can include carbonaceous, calcareous, siliceous, etc.

Grain size is the basis for the classification of clastic sedimentary rocks. Figure 4 is the Udden-Wentworth classification that will be assigned to sedimentary rocks. The individual boundaries are slightly different than the USCS subdivision for soil classification. For field determination of grain sizes, a scale can be used for the coarse grained rocks. For example, the division between siltstone and claystone may not be measurable in the field. The boundary shall be determined by use of a hand lens. If the grains cannot be seen with the naked eye but are distinguishable with a hand lens, the rock is a siltstone. If the grains are not distinguishable with a hand lens, the rock is a claystone.

FIGURE 4

GRAIN SIZE CLASSIFICATION FOR ROCKS

Particle Name	Grain Size Diameter
Cobbles	> 64 mm
Pebbles	4 - 64 mm
Granules	2 - 4 mm
Very Coarse Sand	1 - 2 mm
Coarse Sand	0.5 - 1 mm
Medium Sand	0.25 - 0.5 mm
Fine Sand	0.125 - 0.25 mm
Very Fine Sand	0.0625 - 0.125 mm
Silt	0.0039 - 0.0625 mm

After Wentworth, 1922

5.3.2 Color

The color of a rock can be determined in a similar manner as for soil samples. Rock core samples shall be classified while wet, when possible, and air cored samples shall be scraped clean of cuttings prior to color classifications.

Rock color charts shall not be used unless specified by the Project Manager.

5.3.3 Bedding Thickness

The bedding thickness designations applied to soil classification (see Figure 3) will also be used for rock classification.

5.3.4 Hardness

The hardness of a rock is a function of the compaction, cementation, and mineralogical composition of the rock. A relative scale for sedimentary rock hardness is as follows:

- Soft - Weathered, considerable erosion of core, easily gouged by screwdriver, scratched by fingernail. Soft rock crushes or deforms under pressure of a pressed hammer. This term is always used for the hardness of the saprolite (decomposed rock which occupies the zone between the lowest soil horizon and firm bedrock).

- Medium soft - Slight erosion of core, slightly gouged by screwdriver, or breaks with crumbly edges from single hammer blow.

- Medium hard - No core erosion, easily scratched by screwdriver, or breaks with sharp edges from single hammer blow.

- Hard - Requires several hammer blows to break and has sharp conchoidal breaks. Cannot be scratched with screwdriver.

Note the difference in usage here of the works "scratch" and "gouge." A scratch shall be considered a slight depression in the rock (do not mistake the scraping off of rock flour from drilling with a scratch in the rock itself), while a gouge is much deeper.

5.3.5 Fracturing

The degree of fracturing or brokenness of a rock is described by measuring the fractures or joint spacing. After eliminating drilling breaks, the average spacing is calculated and the fracturing is described by the following terms:

- Very broken (V. BR.) - Less than 2-inch spacing between fractures
- Broken (BR.) - 2-inch to 1-foot spacing between fractures
- Blocky (BL.) - 1- to 3-foot spacing between fractures
- Massive (M.) - 3 to 10-foot spacing between fractures

The structural integrity of the rock can be approximated by calculating the Rock Quality Designation (RQD) of cores recovered. The RQD is determined by adding the total lengths of all pieces exceeding 4 inches and dividing by the total length of the coring run, to obtain a percentage.

Method of Calculating RQD
(After Deere, 1964)

$$RQD\ \% = r/l \times 100$$

r = Total length of all pieces of the lithologic unit being measured, which are greater than 4 inches length, and have resulted from natural breaks. Natural breaks include slickensides, joints, compaction slicks, bedding plane partings (not caused by drilling), friable zones, etc.

l = Total length of the coring run.

5.3.6 Weathering

The degree of weathering is a significant parameter that is important in determining weathering profiles and is also useful in engineering designs. The following terms can be applied to distinguish the degree of weathering:

- Fresh - Rock shows little or no weathering effect. Fractures or joints have little or no staining and rock has a bright appearance.

- Slight - Rock has some staining which may penetrate several centimeters into the rock. Clay filling of joints may occur. Feldspar grains may show some alteration.

- Moderate - Most of the rock, with exception of quartz grains, is stained. Rock is weakened due to weathering and can be easily broken with hammer.

- Severe - All rock including quartz grains is stained. Some of the rock is weathered to the extent of becoming a soil. Rock is very weak.

5.3.7 Other Characteristics

The following items shall be included in the rock description:

- Description of contact between two rock units. These can be sharp or gradational.
- Stratification (parallel, cross stratified).
- Description of any filled cavities or vugs.
- Cementation (calcareous, siliceous, hematitic).
- Description of any joints or open fractures.
- Observation of the presence of fossils.
- Notation of joints with depth, approximate angle to horizontal, any mineral filling or coating, and degree of weathering.

All information shown on the boring logs shall be neat to the point where it can be reproduced on a copy machine for report presentation. The data shall be kept current to provide control of the drilling program and to indicate various areas requiring special consideration and sampling.

5.3.8 Additional Terms Used in the Description of Rock

The following terms are used to further identify rocks:

- Seam - Thin (12 inches or less), probably continuous layer.
- Some - Indicates significant (15 to 40 percent) amounts of the accessory material. For example, rock composed of seams of sandstone (70 percent) and shale (30 percent) would be "sandstone -- some shale seams."
- Few - Indicates insignificant (0 to 15 percent) amounts of the accessory material. For example, rock composed of seam of sandstone (90 percent) and shale (10 percent) would be "sandstone -- few shale seams."
- Interbedded - Used to indicate thin or very thin alternating seams of material occurring in approximately equal amounts. For example, rock composed of thin alternating seams of sandstone (50 percent) and shale (50 percent) would be "interbedded sandstone and shale."
- Interlayered - Used to indicate thick alternating seams of material occurring in approximately equal amounts.

The preceding sections describe the classification of sedimentary rocks. The following are some basic names that are applied to igneous rocks:

- Basalt - A fine-grained extrusive rock composed primarily of calcic plagioclase and pyroxene.
- Rhyolite - A fine-grained volcanic rock containing abundant quartz and orthoclase. The fine-grained equivalent of a granite.
- Granite - A coarse-grained plutonic rock consisting essentially of alkali feldspar and quartz.
- Diorite - A coarse-grained plutonic rock consisting essentially of sodic plagioclase and hornblende.
- Gabbro - A coarse-grained plutonic rock consisting of calcic plagioclase and clinopyroxene. Loosely used for any coarse-grained dark igneous rock.

The following are some basic names that are applied to metamorphic rocks:

- Slate - A very fine-grained foliated rock possessing a well developed slaty cleavage. Contains predominantly chlorite, mica, quartz, and sericite.
- Phyllite - A fine-grained foliated rock that splits into thin flaky sheets with a silky sheen on cleavage surface.
- Schist - A medium to coarse-grained foliated rock with subparallel arrangement of the micaceous minerals which dominate its composition.
- Gneiss - A coarse-grained foliated rock with bands rich in granular and platy minerals.
- Quartzite - A fine- to coarse-grained nonfoliated rock breaking across grains, consisting essentially of quartz sand with silica cement.

5.4 Abbreviations

Abbreviations may be used in the description of a rock or soil. However, they shall be kept at a minimum. Following are some of the abbreviations that may be used:

C	-	Coarse	Lt	-	Light	Yl	-	Yellow
Med	-	Medium	BR	-	Broken	Or	-	Orange
F	-	Fine	BL	-	Blocky	SS	-	Sandstone
V	-	Very	M	-	Massive	Sh	-	Shale
Sl	-	Slight	Br	-	Brown	LS	-	Limestone
Occ	-	Occasional	Bl	-	Black	Fgr	-	Fine-grained
Tr	-	Trace						

5.5 Boring Logs and Documentation

This section describes in more detail the procedures to be used in completing boring logs in the field. Information obtained from the preceding sections shall be used to complete the logs. A sample boring log has been provided as Figure 5.

The field geologist/engineer shall use this example as a guide in completing each boring log. Each boring log shall be fully described by the geologist/engineer as the boring is being drilled. Every sheet contains space for 25 feet of log. Information regarding classification details is provided either on the back of the boring log or on a separate sheet, for field use.

5.5.1 Soil Classification

- Identify site name, boring number, job number, etc. Elevations and water level data to be entered when surveyed data is available.

- Enter sample number (from SPT) under appropriate column. Enter depth sample was taken from (1 block = 1 foot). Fractional footages, i.e., change of lithology at 13.7 feet, shall be lined off at the proportional location between the 13- and 14-foot marks. Enter blow counts (Standard Penetration Resistance) diagonally (as shown). Standard penetration resistance is covered in Section 5.2.3.

- Determine sample recovery/sample length as shown. Measure the total length of sample recovered from the split-spoon sampler, including material in the drive shoe. Do not include cuttings or wash material that may be in the upper portion of the sample tube.

- Indicate any change in lithology by drawing a line at the appropriate depth. For example, if clayey silt was encountered from 0 to 5.5 feet and shale from 5.5 to 6.0 feet, a line shall be drawn at this increment. This information is helpful in the construction of cross-sections. As an alternative, symbols may be used to identify each change in lithology.

- The density of granular soils is obtained by adding the number of blows for the last two increments. Refer to Density of Granular Soils Chart on back of log sheet. For consistency of cohesive soils refer also to the back of log sheet - Consistency of Cohesive Soils. Enter this information under the appropriate column. Refer to Section 5.2.3.

FIGURE 5
COMPLETED BORING LOG (EXAMPLE)

BORING LOG

Page 1 of 1

PROJECT NAME: NSB- SITE
BORING NUMBER: SB/MW 1
PROJECT NUMBER: 9594
DATE: 3/8/96
DRILLING COMPANY: SOILTEST CO.
GEOLOGIST: SJ CONTI
DRILLING RIG: CME-55
DRILLER: R. ROCK

Sample No. and Type or RQD	Depth (Ft.) or Run No.	Blows / 6" or RQD (%)	Sample Recovery / Sample Length	Lithology Change (Depth/Ft.) or Screened Interval	MATERIAL DESCRIPTION: Soil Density/ Consistency or Rock Hardness	Color	Material Classification	USCS*	Remarks	PID/FID Reading (ppm): Sample	Sampler BZ	Borehole**	Driller BZ**
S-1	0.0	7/6	1.5/2.0		M DENSE	BRN	SILTY SAND - SOME	SM	MOIST SL. ORG.	5	0	0	0
@ 0800	2.0	9/10				TO BLK	ROCK FR. - TR BRICKS		ODOR				
							(FILL)		FILL TO 4'±				
	4.0			4.0									
S-2 @		5/7	2.0/2.0		M DENSE	BRN	SILTY SAND - TR FINE	SM	MOIST - W ODOR	10	0	-	-
0810	6.0	9/8					GRAVEL		NAT. MATL. TOOK SAMPLE				
	•			7' ▽ ±					SB01-0406 FOR ANALYSIS				
	8.0			8.0									
S-3 @		6/8	1.9/2.0		DENSE	TAN BRN	FINE TO COARSE SAND	SW	WET	0	0	0	0
0820	10.0	17/16					TR. F. GRAVEL		HIT WATER ≈ 7'±				
	12.0			12.0									
S-4 @		7/6	1.6/2.0		STIFF	GRAY	SILTY CLAY	CL	MOIST → WET	0	.5	-	-
0830	14.0	5/8											
	15.0			15.0					AUGER REF @ 15'				
				[illegible]	M HARD	BRN	SILTSTONE	WBR	WEATHERED				
				16					LO ∠ JNTS @ 15.5				
9/5	①		4.0/5.0						WATER STAINS @ 16.5, 17.1, 17.5	0	0	0	0
				19'					LOSING SOME				
	20.0				HARD	GRAY	SANDSTONE - SOME	BR	DRILL H2O @ 17'±				
							SILTSTONE		SET TEMP 6" CAS TO 15.5				
4.9/5.0	②		5.0/5.0						SET 2"Φ PVC SCREEN 16-25	0	0	0	0
									SAND 14-25				
	25.0			25					PELLETS 12-14				

* When rock coring, enter rock brokeness.
** Include monitor reading in 6 foot intervals @ borehole. Increase reading frequency if elevated response read.

• 1-2OZ
1-8OZ

Drilling Area Background (ppm): 0

Remarks: CME-55 RIG, 4 1/4" ID HSA - 9" OD ±
2" SPLIT SPOONS - 140 LB HAMMER - 30" DROP
NX CORE IN BEDROCK RUN ① = 25 min, RUN ② = 15 min

Converted to Well: Yes ✓ No ______ Well I.D. #: MW-1

- Enter color of the material in the appropriate column.

- Describe material using the USCS. Limit this column for sample description only. The predominant material is described last. If the primary soil is silt but has fines (clay) - use clayey silt. Limit soil descriptors to the following:

  - Trace: 0 - 10 percent
  - Some: 11 - 30 percent
  - And/Or: 31 - 50 percent

- Also indicate under Material Classification if the material is fill or natural soils. Indicate roots, organic material, etc.

- Enter USCS symbol - use chart on back of boring log as a guide. If the soils fall into one of two basic groups, a borderline symbol may be used with the two symbols separated by a slash. For example ML/CL or SM/SP.

- The following information shall be entered under the "Remarks" column and shall include, but is not limited by, the following:

  - Moisture - estimate moisture content using the following terms - dry, moist, wet and saturated. These terms are determined by the individual. Whatever method is used to determine moisture, be consistent throughout the log.

  - Angularity - describe angularity of coarse grained particles using the terms angular, subangular, subrounded, or rounded. Refer to ASTM D 2488 or Earth Manual for criteria for these terms.

  - Particle shape - flat, elongated, or flat and elongated.

  - Maximum particle size or dimension.

  - Water level observations.

  - Reaction with HCl - none, weak, or strong.

- Additional comments:

  - Indicate presence of mica, caving of hole, when water was encountered, difficulty in drilling, loss or gain of water.

  - Indicate odor and Photoionization Detector (PID) or Flame Ionization Detector (FID) reading if applicable.

  - Indicate any change in lithology by drawing a line through the lithology change column and indicate the depth. This will help when cross-sections are subsequently constructed.

  - At the bottom of the page indicate type of rig, drilling method, hammer size and drop, and any other useful information (i.e., borehole size, casing set, changes in drilling method).

- Vertical lines shall be drawn (as shown in Figure 5) in columns 6 to 8 from the bottom of each sample to the top of the next sample to indicate consistency of material from sample to sample, if the material is consistent. Horizontal lines shall be drawn if there is a change in lithology, then vertical lines drawn to that point.

- Indicate screened interval of well, as needed, in the lithology column. Show top and bottom of screen. Other details of well construction are provided on the well construction forms.

5.5.2 Rock Classification

- Indicate depth at which coring began by drawing a line at the appropriate depth. Indicate core run depths by drawing coring run lines (as shown) under the first and fourth columns on the log sheet. Indicate RQD, core run number, RQD percent, and core recovery under the appropriate columns.

- Indicate lithology change by drawing a line at the appropriate depth as explained in Section 5.5.1.

- Rock hardness is entered under designated column using terms as described on the back of the log or as explained earlier in this section.

- Enter color as determined while the core sample is wet; if the sample is cored by air, the core shall be scraped clean prior to describing color.

- Enter rock type based on sedimentary, igneous or metamorphic. For sedimentary rocks use terms as described in Section 5.3. Again, be consistent in classification. Use modifiers and additional terms as needed. For igneous and metamorphic rock types use terms as described in Sections 5.3.8.

- Enter brokenness of rock or degree of fracturing under the appropriate column using symbols VBR, BR, BL, or M as explained in Section 5.3.5 and as noted on the back of the Boring Log.

- The following information shall be entered under the remarks column. Items shall include but are not limited to the following:

  - Indicate depths of joints, fractures and breaks and also approximate to horizontal angle (such as high, low), i.e., 70° angle from horizontal, high angle.
  - Indicate calcareous zones, description of any cavities or vugs.
  - Indicate any loss or gain of drill water.
  - Indicate drop of drill tools or change in color of drill water.

- Remarks at the bottom of Boring Log shall include:

  - Type and size of core obtained.
  - Depth casing was set.
  - Type of rig used.

- As a final check the boring log shall include the following:

  - Vertical lines shall be drawn as explained for soil classification to indicate consistency of bedrock material.

  - If applicable, indicate screened interval in the lithology column. Show top and bottom of screen. Other details of well construction are provided on the well construction forms.

5.5.3 Classification of Soil and Rock from Drill Cuttings

The previous sections describe procedures for classifying soil and rock samples when cores are obtained. However, some drilling methods (air/mud rotary) may require classification and borehole logging based on identifying drill cuttings removed from the borehole. Such cuttings provide only general information on subsurface lithology. Some procedures that shall be followed when logging cuttings are:

- Obtain cutting samples at approximately 5-foot intervals, sieve the cuttings (if mud rotary drilling) to obtain a cleaner sample, place the sample into a small sample bottle or "zip lock" bag for future reference, and label the jar or bag (i.e. hole number, depth, date, etc.). Cuttings shall be closely examined to determine general lithology.

- Note any change in color of drilling fluid or cuttings, to estimate changes in lithology.

- Note drop or chattering of drilling tools or a change in the rate of drilling, to determine fracture locations or lithologic changes.

- Observe loss or gain of drilling fluids or air (if air rotary methods are used), to identify potential fracture zones.

- Record this and any other useful information onto the boring log as provided in Figure 1.

This logging provides a general description of subsurface lithology and adequate information can be obtained through careful observation of the drilling process. It is recommended that split-barrel and rock core sampling methods be used at selected boring locations during the field investigation to provide detailed information to supplement the less detailed data generated through borings drilled using air/mud rotary methods.

5.6 Review

Upon completion of the borings logs, copies shall be made and reviewed. Items to be reviewed include:

- Checking for consistency of all logs.
- Checking for conformance to the guideline.
- Checking to see that all information is entered in their respective columns and spaces.

6.0 REFERENCES

Unified Soil Classification System (USCS).

ASTM D2488, 1985.

Earth Manual, U.S. Department of the Interior, 1974.

7.0 RECORDS

Originals of the boring logs shall be retained in the project files.

TETRA TECH NUS, INC.	**STANDARD OPERATING PROCEDURES**	Number GH-2.5	Page 1 of 9
		Effective Date 06/99	Revision 1
		Applicability Tetra Tech NUS, Inc.	
		Prepared Earth Sciences Department	
Subject GROUNDWATER CONTOUR MAPS AND FLOW DETERMINATIONS		Approved D. Senovich	

TABLE OF CONTENTS

1.0 PURPOSE

The purpose of this procedure is to provide a basic understanding of developing contour maps and the approaches used to identify and quantify the direction and rate of groundwater flow and contaminant plume movement.

2.0 SCOPE

This procedure provides only a general overview of the field techniques, mathematical and physical relationships and data handling procedures used for determining groundwater flow direction and rate. The references identified herein can provide a more complete explanation of particular methods cited, as well as a more comprehensive discussion on the interpretation of hydrogeologic data.

3.0 GLOSSARY

Aquifer - A geologic formation capable of transmitting usable quantities of groundwater to a well or other discharge point.

Aquitard - A geologic formation which retards the flow of groundwater due to its low permeability.

Confined Aquifer - An aquifer that is overlain and underlain by zones of lower permeability (aquitards). If the aquifer is "artesian," the potentiometric head of the aquifer at a given point is higher than the top of the zone comprising the aquifer at that point.

Equipotential Line - A line connecting points of equal elevation of the water table or potentiometric surface. Equipotential lines on the water table are also called water table contour lines.

Flow Line - A flow line indicates the direction of groundwater movement within the saturated zone. Flow lines are drawn perpendicular to equipotential lines.

Flow Net - A diagram of groundwater flow showing flow lines and equipotential lines.

Hydraulic Conductivity (K) - A quantitative measure of the ability of porous material to transmit water. Volume of water that will flow through a unit cross sectional area of porous material per unit time under a head gradient. Hydraulic conductivity is dependent upon properties of the medium and fluid.

Hydraulic Gradient (i) - The rate of change of hydraulic head per unit distance of flow at a given point and in the downgradient direction.

Hydraulic Head - The height to which water will rise inside a well casing, equal to the elevation head plus the pressure head. In a well screened across the water table, hydraulic head equals the elevation head, as the pressure head equals 0. In wells screened below the water table in an unconfined aquifer or screened at any interval within a confined aquifer, the head is the sum of the elevation of the aquifer (the elevation head) and the fluid pressure of the water confined in the aquifer (the pressure head).

Potentiometric (piezometric) Surface - A hypothetical surface that coincides with the static level of the water in an aquifer (i.e., the maximum elevation to which water will rise in a well or piezometer penetrating the aquifer). The term "potentiometric surface" is usually applied to confined aquifers, although the water table is the potentiometric surface of an unconfined aquifer.

Unconfined Aquifer - An aquifer in which the water table forms the upper boundary.

Water Table - The surface in the groundwater system at which the fluid pressure is equal to atmospheric pressure (i.e., the net pressure head is zero) and below which all strata are saturated with water.

4.0 RESPONSIBILITIES

Project Hydrogeologist - The project hydrogeologist has overall responsibility for obtaining water level measurements and developing groundwater contour maps. The hydrogeologist (with the concurrence of the Project Manager) shall specify the reference point from which water levels are measured (usually a specific point on the upper edge of the inner well casing), the number of data points needed and which wells shall be used for a contour map, and how many complete sets of water levels are required to adequately define groundwater flow directions (e.g., if there are seasonal variations).

Field Personnel - All supporting field personnel must have a basic familiarity with the equipment and procedures involved in obtaining water levels, and must be aware of any project-specific requirements.

5.0 PROCEDURES

5.1 Potentiometric Surface Mapping

5.1.1 Selection of Wells

All wells used to prepare a flow net in a plan or map view should represent the same hydrogeologic unit, be it aquifer or aquitard. All water level measurements used shall be collected on the same day, preferably within 2-3 hours. This is especially important when working in an area where groundwater levels are tidally influenced or influenced by pumping.

The recorded water levels, monitoring-well construction data, site geology, and topographic setting must be reviewed to ascertain that the wells are completed in the same hydrogeologic unit and to determine if strong vertical hydraulic gradients may be present. Such conditions will be manifested by a pronounced correlation between well depth and water level, or by a difference in water level between two wells located near each other but set to different depths or having different screen lengths. Professional judgment of the hydrogeologist is important in this determination. If vertical gradients are significant, the data to be used must be limited vertically, and only wells finished in a chosen vertical zone of the hydrogeologic unit can be used.

At least three wells must be used to provide an estimation of the direction of groundwater flow; information from many more wells are needed to provide an accurate contour map. Generally, shallow systems require data from more wells than deep systems for accurate contour mapping. Potentiometric surface mapping for shallow flow systems also requires water level measurements from nearby surface water bodies.

5.1.2 Water Level Measurements

After selection of the wells to be used for mapping, the next step in determining the direction of groundwater flow is to obtain water level elevations from the selected points. In addition, any other readily available wells/surface water bodies should be measured to ensure that sufficient data are available for interpretation purposes.

Elevations are obtained from measurements of the depth to water in a monitoring well or piezometer taken from the top of the well casing (see SOP GH-1.2) and then referencing the elevation of the casing to a chosen and consistent datum point, usually mean sea level. Subtracting the depth to water from the casing elevation provides the elevation of the potentiometric surface. Elevations of points and areas of groundwater discharge or recharge such as springs, seeps, streams, rivers, and lakes also need to be determined, typically through staff gauge measurements. Comparison of these elevations, which represent hydraulic heads, will reveal the direction of flow because groundwater flows from areas of high head to areas of low head.

5.1.3 Construction of Equipotential Lines

Graphical methods available for depicting the flow of groundwater include the use of equipotential lines and flow lines to construct potentiometric surface maps and vertical flow nets. If the hydrogeologic system consists of a water table aquifer and one or more confined aquifers, separate contour maps should be prepared for each aquifer system. Water table maps should be developed using water level measurements obtained from monitoring wells screened at the unsaturated-saturated interface. Water level measurements collected from monitoring wells screened in the deeper portions of an unconfined aquifer should generally be contoured as a separate potentiometric surface map. Surface water discharge or recharge features are contoured in the water table system. Vertical flow nets should be constructed using a cross section aligned parallel to the direction of groundwater flow. All water level measurements along this cross section, both deep and shallow, are used in developing equipotential lines and flow lines for the flow net.

To construct equipotential lines, water level elevations in the chosen wells are plotted on a site map. Other hydrogeologic features associated with the zone of interest -- such as seeps, wetlands, and surface-water bodies -- should also be plotted along with their elevations.

The data should then be contoured, using mathematically valid and generally accepted techniques. Linear interpolation is the most commonly used technique. However, quadratic interpolation or any technique of trend-surface analysis or data smoothing is acceptable. Computer-generated contour maps may be useful rough mapping of large data sets; however, final, detailed mapping must <u>always</u> be performed by hand by an experienced hydrogeologist. Contour lines shall be drawn as smooth, continuous lines which never cross one another.

Inspect the contour map, noting known features, such as pumping wells and site topography. The contour lines must be adjusted utilizing the professional judgment of the hydrogeologist in accordance with these features. Closed contours should be avoided unless a known groundwater sink (i.e., pumping well) or mound exists. Groundwater mounding is common under landfills and lagoons; if the data imply this, the feature must be evident in the contour plot.

5.1.4 Determination of Groundwater-Flow Direction

Flow lines shall be drawn so that they are perpendicular to equipotential lines. Flow lines will begin at high head elevations and end at low head elevations. Closed highs will be the source of additional flow lines. Closed depressions (i.e., wells) will be the termination of some flow lines. Care must be used in areas with significant vertical gradients to avoid erroneous conclusions concerning gradients and flow directions.

5.2 Groundwater Flow Considerations

Groundwater movement is an integral part of the hydrologic cycle. Recharge to the shallow groundwater environment generally occurs by infiltration of precipitation through an upper unsaturated soil zone. Movement is downward under the force of gravity until the water reaches the saturated zone of the water table aquifer. Once water is part of the water table aquifer, movement is controlled by differences in hydraulic head, with movement from areas of high head to areas of low head. Areas of low head include natural discharge areas such as springs, lakes, rivers, and, ultimately, the ocean. These features can be considered as outcrops of the water table. Points of low head also are created by pumping wells.

Local head differences and consequent vertical flow patterns within an aquifer can be detected by well clusters. A well cluster consists of several adjacent wells, generally installed within a few feet of each other, and screened at different depths. Variations in water levels in these closely spaced wells indicates the vertical component of groundwater flow within an aquifer, provided that the wells are all screened within the same aquifer.

The number, location, and extent of geologic units and their properties with regard to aquifer or aquitard characteristics must be understood to properly interpret water level data gathered from the monitoring system. This firm understanding of the hydrogeologic system must be developed through a program of borings, wells, and interpretation of subsurface geology. The adequacy of the positions and depths of borings/wells used to define relevant subsurface hydrogeologic conditions must also be assessed. The location of surface water discharge or recharge points must be considered. Surface water features influence the system, as flow is most likely toward them (if they are discharge points) or away from them (if they are recharge points). Man-made discharge or recharge features such as pumping or injection wells, ditches, and trenches can also affect the flow of groundwater.

5.3 Determination of Flow Rate

Darcy's Law states that the quantity of water flowing through a geologic material is dependent upon the permeability of the material, the hydraulic gradient, and the cross sectional area through which the water flows. This relation is expressed in the equation:

$$Q = KiA$$

where:

Q	=	volume of water flowing through the cross sectional area of the formation (L^3/T).
K	=	hydraulic conductivity (L/T).
i	=	hydraulic gradient (L/L, i.e., dimensionless).
A	=	cross sectional area of formation being considered (L^2).

The relation is similar to one used in stream flow measurements where:

$$Q = VA$$

where:

Q	=	discharge from the cross sectional area of a stream or pipe (L^3/T).
V	=	average velocity of flowing water (L/T).
A	=	cross sectional area through which water flows (L^2).

The velocity of water movement in a geologic formation depends on the specific formation properties and the head differences across the formation. This relation is defined in the equation:

$$V = \frac{Ki}{n}$$

where:

V	=	average linear velocity of groundwater through the formation (L/T)
K	=	hydraulic conductivity (L/T)
i	=	hydraulic gradient (dimensionless)
n	=	porosity (expressed as a fraction).

Values of porosity for several geologic materials are given in Attachment A. More accurate and specific values of porosity can be obtained by laboratory analysis of a formation sample or from an unconfined aquifer pumping test.

Hydraulic conductivity is related to the permeability of the formation and depends on the size and interconnection of the pore spaces. In isotropic and homogeneous formations, the hydraulic conductivity will be the same vertically and horizontally. In anisotropic formations, horizontal and vertical conductivity can be markedly different and the vertical hydraulic conductivity can be up to several orders of magnitude lower than the horizontal hydraulic conductivity. Typically, most formations are anisotropic with horizontal hydraulic conductivities at least several times as high as the vertical hydraulic conductivities.

Generally, hydraulic conductivities are high for sands, gravels, and limestone containing large solution cavities and low for silts, clays, and tightly fractured rock. Attachment A gives values of hydraulic conductivity for several geologic materials. More accurate values can be obtained during field testing of aquifers or from laboratory measurements on undisturbed cores. Results from field testing usually provide higher (and more representative) hydraulic conductivities than laboratory testing because full-scale field testing includes the effects of the formational macrostructure (i.e., secondary permeability due to jointing or fractures) which is not reflected in the testing of a small sample in the laboratory.

The hydraulic gradient, i, is determined from field measurements of hydraulic head obtained from water level measuring points. Do <u>not</u> measure gradient from well to well; measure across equipotential lines that are drawn based on the well (and other) data. Once a potentiometric surface map has been generated using the hydraulic head data, the hydraulic gradient can be calculated using the following formula:

$$i = \frac{dh}{dl}$$

where:

dh	=	change in head (L)
dl	=	distance between equipotential lines (L)

The hydraulic gradient along any flow line can be calculated from a potentiometric surface map by dividing the change in head by the length of the flow line, typically beginning and ending at equipotential lines. The longer the distance over which the head change is measured, the more representative the gradient is of overall conditions.

When chemical solutes are traveling in groundwater, as in cases of groundwater contamination, the calculated groundwater velocity may predict migration rates in excess of what is actually observed. The difference in chemical versus water velocities may be due to attenuation or biodegradation of the chemical species in the aquifer. Attenuation is most often caused by adsorption of the chemical contaminant onto the formation grains or matrix. The result is that the chemical does not appear at the downgradient sampling point as quickly as the velocity calculation predicts. An equation to correct for this attenuation is:

$$V_c = V_w / \left(1 + K_d P_b / n\right)$$

where:

V_c	=	velocity of the chemical solute flow (L/T)
V_w	=	velocity of groundwater flow (L/T)
P_b	=	formation mass bulk density (M/L^3)
n	=	formation porosity (expressed as a fraction)
K_d	=	distribution coefficient = (L^3/M)

The K_d is equal to the mass of solute per unit mass of solid phase divided by the concentration of solute in solution. The term in the denominator is known as the retardation factor.

Density and/or viscosity differences between water and contaminants can also cause velocity determination errors. Light hydrocarbons such as gasoline are less dense than water and consequently float on the water table. These contaminants can migrate along the water table surface at rates faster or slower than the rate of groundwater movement, depending on specific conditions, and may also volatilize into unsaturated soil pore spaces. Oils are more viscous than water and will typically migrate more slowly due to the viscosity difference. Contaminants denser than water such as heavy hydrocarbons (e.g., coal tar) or chlorinated compounds (e.g., TCE, PCE) tend to sink to the bottom of an aquifer if present in concentrations exceeding their solubility limit (these chemicals are often referred to as dense, nonaqueous phase liquids, or DNAPLs if present as a separate-phase liquid). Here, the contamination may move at faster or slower rates than the overlying groundwater or may actually move in a direction opposite to that of the groundwater, depending on the geologic characteristics of the aquifer base and direction of dip of the underlying aquitard.

Other factors involving the physicochemical interaction between the chemical and the groundwater, such as dilution (mixing contaminated water or chemicals with additional quantities of groundwater) and dispersion (molecular diffusion of the chemical throughout the groundwater regime), can also affect the observed rates of travel of contaminants in groundwater. In addition to such physicochemical characteristics, all of the aquifer and aquitard properties and groundwater flow characteristics described above must be known so that adequate and accurate estimations of the extent and rate of groundwater contaminant migration can be developed.

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ATTACHMENT A

GENERALIZED POROSITY AND HYDRAULIC CONDUCTIVITY VALUES FOR GEOLOGIC MATERIALS

Material	Porosity Range (%)	Hydraulic Conductivity Range	
		cm/sec	ft/day
Gravel	30-40	10^{-1} to 10^{-2}	280 to 2.8×10^5
Coarse sand (clean)	30-40	10^{-1} to 1	280 to 2,800
Medium sand (clean)	35-45	10^{-2} to 10^{-1}	28 to 280
Fine sand (clean)	40-50	5×10^{-4} to 10^{-2}	1.4 to 28
Silty sand	25-40	10^{-5} to 10^{-2}	0.03 to 280
Glacial Till	Variable	10^{-10} to 10^{-4}	3×10^{-7} to 0.3
Unweathered Clay/Shale	45-55 (clay)	10^{-7} to 10^{-4}	3×10^{-4} to 0.3 (horizontal)
		10^{-10} to 10^{-6}	3×10^{-7} to 3×10^{-3} (vertical)
Karst Limestone	---	10^{-4} to 10^{-1}	0.3 to 2,800
Fractured Igneous/Metamorphic Rocks	---	10^{-6} to 10^{-1}	3×10^{-3} to 280
Sandstone	5-30	10^{-8} to 10^{-4}	3×10^{-5} to 0.3

Source: References 1 and 2

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		Applicability: Tetra Tech NUS, Inc.	
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1.0 PURPOSE

This procedure provides general guidance and information pertaining to proper monitoring well design, installation, and development.

2.0 SCOPE

This procedure is applicable to the construction of monitoring wells. The methods described herein may be modified by project-specific requirements for monitoring well construction. In addition, many regulatory agencies have specific regulations pertaining to monitoring well construction and permitting. These requirements must be determined during the project planning phases of the investigation, and any required permits must be obtained before field work begins. Innovative monitoring well installation techniques, which typically are not used, will be discussed only generally in this procedure.

3.0 GLOSSARY

Monitoring Well - A well which is screened, cased, and sealed which is capable of providing a groundwater level and groundwater sample representative of the zone being monitored. Some monitoring wells may be constructed as open boreholes.

Piezometer - A pipe or tube inserted into the water bearing zone, typically open to water flow at the bottom and to the atmosphere at the top, and used to measure water level elevations. Piezometers may range in size from 1/2-inch-diameter plastic tubes to well points or monitoring wells.

Potentiometric Surface - The surface representative of the level to which water will rise in a well cased to the screened aquifer.

Well Point (Drive Point) - A screened or perforated tube (Typically 1-1/4 or 2 inches in diameter) with a solid, conical, hardened point at one end, which is attached to a riser pipe and driven into the ground with a sledge hammer, drop weight, or mechanical vibrator. Well points may be used for groundwater injection and recovery, as piezometers (i.e., to measure water levels) or to provide groundwater samples for water quality data.

4.0 RESPONSIBILITIES

Driller - The driller provides adequate and operable equipment, sufficient quantities of materials, and an experienced and efficient labor force capable of performing all phases of proper monitoring well installation and construction. The driller may also be responsible for obtaining, in advance, any required permits for monitoring well installation and construction.

Field Geologist - The field geologist supervises and documents well installation and construction performed by the driller, and insures that well construction is adequate to provide representative groundwater data from the monitored interval. Geotechnical engineers, field technicians, or other suitable trained personnel may also serve in this capacity.

5.0 PROCEDURES

5.1 Equipment/Items Needed

Below is a list of items that may be needed when installing a monitoring well or piezometer:

- Health and safety equipment (hard hats, safety glasses, etc.) as required by the Site Safety Officer.
- Well drilling and installation equipment with associated materials (typically supplied by the driller).
- Hydrogeologic equipment (weighted engineer's tape, water level indicator, retractable engineers rule, electronic calculator, clipboard, mirror and flashlight - for observing downhole activities, paint and ink marker for marking monitoring wells, sample jars, well installation forms, and a field notebook).
- Drive point installation tools (sledge hammer, drop hammer, or mechanical vibrator; tripod, pipe wrenches, drive points, riser pipe, and end caps).

5.2 Well Design

The objectives and intended use for each monitoring well must be clearly defined before the monitoring system is designed. Within the monitoring system, different monitoring wells may serve different purposes and, therefore, require different types of construction. During all phases of the well design, attention must be given to clearly documenting the basis for design decisions, the details of well construction, and the materials used. The objectives for installing the monitoring wells may include:

- Determining groundwater flow directions and velocities.
- Sampling or monitoring for trace contaminants.
- Determining aquifer characteristics (e.g., hydraulic conductivity).

Siting of monitoring wells shall be performed after a preliminary estimation of the groundwater flow direction. In most cases, groundwater flow directions and potential well locations can be determined by an experienced hydrogeologist through the review of geologic data and the site terrain. In addition, data from production wells or other monitoring wells in the area may be used to determine the groundwater flow direction. If these methods cannot be used, piezometers, which are relatively inexpensive to install, may have to be installed in a preliminary investigative phase to determine groundwater flow direction.

5.2.1 Well Depth, Diameter, and Monitored Interval

The well depth, diameter, and monitored interval must be tailored to the specific monitoring needs of each investigation. Specification of these items generally depends on the purpose of the monitoring system and the characteristics of the hydrogeologic system being monitored. Wells of different depth, diameter, and monitored interval can be employed in the same groundwater monitoring system. For instance, varying the monitored interval in several wells, at the same location (cluster wells) can help to determine the vertical gradient and the depths at which contaminants are present. Conversely, a fully penetrating well is usually not used to quantify or vertically locate a contaminant plume, since groundwater samples collected in wells that are screened over the full thickness of the water-bearing zone will be representative of average conditions across the entire monitored interval. However, fully penetrating wells can be used to establish the existence of contamination in the water-bearing zone. The well diameter desired depends upon the hydraulic characteristics of the water-bearing zone, sampling requirements, drilling method and cost.

The decision concerning the monitored interval and well depth is based on the following (and possibly other) information:

- The vertical location of the contaminant source in relation to the water-bearing zone.
- The depth, thickness and uniformity of the water-bearing zone.
- The anticipated depth, thickness, and characteristics (e.g., density relative to water) of the contaminant plume.
- Fluctuation in groundwater levels (due to pumping, tidal influences, or natural recharge/discharge events).
- The presence and location of contaminants encountered during drilling.
- Whether the purpose of the installation is for determining existence or non-existence of contamination or if a particular stratigraphic zone is being investigated.
- The analysis of borehole geophysical logs.

In most situations where groundwater flow lines are horizontal, depending on the purpose of the well and the site conditions, monitored intervals are 20 feet or less. Shorter screen lengths (5 feet or less) are usually required where flow lines are not horizontal, (i.e., if the wells are to be used for accurate measurement of the potentiometric head at a specific point).

Many factors influence the diameter of a monitoring well. The diameter of the monitoring well depends on the application. In determining well diameter, the following needs must be considered:

- Adequate water volume for sampling.
- Drilling methodology.
- Type of sampling device to be used.
- Costs.

Standard monitoring well diameters are 2, 4, 6, or 8 inches. Drive points are typically 1-1/4 or 2 inches in diameter. For monitoring programs which require screened monitoring wells, either a 2-inch or 4-inch-diameter well is preferred. Typically, well diameters greater than 4 inches are used in monitoring programs in which open-hole bedrock monitoring wells are used. With smaller diameter wells, the volume of stagnant water in the well is minimized, and well construction costs are reduced; however, the sampling devices that can be used are limited.

In specifying well diameter, sampling requirements must be considered (up to a total of 4 gallons of water may be required for a single sample to account for full organic and inorganic analyses, and split samples), particularly if the monitored formation is known to be a low-yielding formation. The unit volume of water contained within a monitoring well is dependent on the well diameter as follows:

Casing Inside Diameter (Inch)	Standing Water Length to Obtain 1 Gallon Water (Feet)
2	6.13
4	1.53
6	0.68

If a well recharges quickly after purging, then well diameter may not be an important factor regarding sample volume requirements.

Pumping tests for determining aquifer characteristics may require larger diameter wells (for installation of high capacity pumps); however, in small-diameter wells in-situ permeability tests can be performed during drilling or after well installation is completed.

5.2.2 Riser Pipe and Screen Materials

Well materials are specified by diameter, type of material, and thickness of pipe. Well screens require an additional specification of slot size. Thickness of pipe is referred to as "Schedule" for polyvinyl chloride (PVC) casing and is usually Schedule 40 (thinner wall) or 80 (thicker wall). Steel pipe thickness is often referred to as "Strength". Standard Strength is usually adequate for monitoring well purposes. With larger diameter pipe, the wall thickness must be greater to maintain adequate strength. The required thickness is also dependent on the method of installation; risers for drive points require greater strength than wells installed inside drilled borings.

The selection of well screen and riser materials depends on the method of drilling, the type of subsurface materials the well penetrates, the type of contamination expected, and natural water quality and depth. Cost and the level of accuracy required are also important. The materials generally available are Teflon, stainless steel, PVC galvanized steel, and carbon steel. Each has advantages and limitations (see Attachment A of this guideline for an extensive presentation on this topic). The two most commonly used materials are PVC and stainless steel. Properties of these two materials are compared in Attachment B. Stainless steel is a good choice where trace metals or organic sampling is required; however, costs are high. Teflon materials are extremely expensive, but are relatively inert and provide the least opportunity for water contamination due to well materials. PVC has many advantages, including low cost, excellent availability, light weight, ease of manipulation, and widespread acceptance. The crushing strength of PVC may limit the depth of installation, but the use of Schedule 80 materials may overcome some of the problems associated with depth. However, the smaller inside diameter of Schedule 80 pipe may be an important factor when considering the size of bailers or pumps required for sampling or testing. Due to this problem, the minimum well pipe size recommended for Schedule 80 wells is 4-inch I.D.

Screens and risers may have to be decontaminated before use because oil-based preservatives and oil used during thread cutting and screen manufacturing may contaminate samples. Metal pipe may corrode and release metal ions or chemically react with organic constituents, but this is considered a minor issue. Galvanized steel is not recommended where samples may be collected for metals analyses, as zinc and cadmium levels in groundwater samples may become elevated from leaching of the zinc coating.

Threaded, flush-joint casing is most often preferred for monitoring well applications. PVC, Teflon, and steel can all be obtained with threaded joints. Welded-joint steel casing is also acceptable. Glued PVC may release organic contaminants into the well, and therefore, should not be used if the well is to be sampled for organic constituents.

When the water-bearing zone is in consolidated bedrock, such as limestone or fractured granite, a well screen is often not necessary (the well is simply an open hole in bedrock). Unconsolidated materials, such as sands, clay, and silts require a screen. A screen slot size of 0.010 or 0.020 inch is generally used when a screen is necessary, and the annular borehole space around the screened interval is artificially packed with an appropriately sized sand, selected based on formation grain size. The slot size controls the quantity of water entering the well and prevents entry of natural materials or sand pack. The screen shall pass no more than 10 percent of the pack material, or in-situ aquifer material. The site geologist shall specify the combination of screen slot size and sand pack which will be compatible with the water-bearing zone, to maximize groundwater inflow and minimize head losses and movement of fines into the wells. For example, as a standard procedure, a Morie No. 1 or No. 10 to No. 20 U.S. Standard Sieve size filter pack is typically appropriate for a 0.020-inch slot screen; however, a No. 20 to No. 40 U.S. Standard Sieve size filter pack is typically appropriate for a 0.010-inch slot screen.

5.2.3 Annular Materials

Materials placed in the annular space between the borehole and riser pipe and screen include a sand pack when necessary, a bentonite seal, and cement-bentonite grout. The sand pack is usually a medium- to coarse-grained poorly graded, silica sand and should relate to the grain size of the aquifer sediments. The quantity of sand placed in the annular space is dependent upon the length of the screened interval, but should always extend at least 1 foot above the top of the screen. At least 1 to 3 feet of bentonite pellets or equivalent shall be placed above the sand pack. Cement-bentonite grout (or equivalent) is then placed to extent from the top of the bentonite pellets to the ground surface.

On occasion, and with the concurrence of the involved regulatory agencies, monitoring wells may be packed naturally (i.e., no artificial sand pack installed). In this case, the natural formation material is allowed to collapse around the well screen after the well is installed. This method has been used where the formation material itself is a relatively uniform grain size, or when artificial sand packing is not possible due to borehole collapse.

Bentonite expands by absorbing water and provides a seal between the screened interval and the overlying portion of the annular space and formation. Cement-bentonite grout is placed on top of the bentonite pellets, extending to the surface. The grout effectively seals the remaining borehole annulus and eliminates the possibility for surface infiltration reaching the screened interval. Grouting also replaces material removed during drilling and prevents hole collapse and subsidence around the well. A tremie pipe should be used to introduce grout from the bottom upward, to prevent bridging, and to provide a better seal. In shallow boreholes that don't collapse, it may be more practical to pour the grout from the surface without a tremie pipe.

Grout is a general term which has several different connotations. For all practical purposes within the monitoring well installation industry, grout refers to the solidified material which is installed and occupies the annular space above the bentonite pellet seal. Grout, most of the time, is made up of one or two assemblages of material, (e.g., cement and/or bentonite). A cement-bentonite grout, which is the most common type of grout used in monitoring well completions, normally is a mixture of cement, bentonite, and water at a ratio of one 90-pound bag of Portland Type I cement, plus 3 to 5 pounds of granular or flake-type bentonite, and 6-7 gallons of water. A neat cement consists of one ninety-pound bag of Portland Type I cement and 6-7 gallons of water. A bentonite slurry (bentonite and water mixed to a thick but pumpable mixture) is sometimes used instead of grout for deep well installations where placement of bentonite pellets is difficult. Bentonite chips are also occasionally used for annular backfill in place of grout.

In certain cases, the borehole may be drilled to a depth greater than the anticipated well installation depth. For these cases, the well shall be backfilled to the desired depth with bentonite pellets/chips or sand. A short (1- to 2-foot) section of capped riser pipe sump is sometimes installed immediately below the screen, as a silt reservoir, when significant post-development silting is anticipated. This will ensure that the entire screen surface remains unobstructed.

5.2.4 Protective Casing

When the well is completed and grouted to the surface, a protective steel casing is typically placed over the top of the well. This casing generally has a hinged cap and can be locked to prevent vandalism. The protective casing has a larger diameter than the well and is set into the wet cement grout over the well upon completion. In addition, one hole is drilled just above the cement collar through the protective casing which acts as a weep hole for the flow of water which may enter the annulus during well development, purging, or sampling.

A protective casing which is level with the ground surface (flush-mounted) is used in roadway or parking lot applications where the top of a monitoring well must be below the pavement. The top of the riser pipe is placed 4 to 5 inches below the pavement, and a locking protective casing is cemented in place to 3 inches below the pavement. A large diameter, manhole-type protective collar is set into the wet cement around the well with the top set level with or slightly above the pavement. An appropriately-sized id is placed over the protective sleeve. The cement should be slightly mounded to direct pooled water away from the well head.

5.3 Monitoring Well Installation

Pertinent data regarding monitoring well installation shall be recorded on log sheets as depicted and discussed in SOP SA-6.3. Attachments to this referenced SOP illustrate terms and physical construction of various types of monitoring wells.

5.3.1 Monitoring Wells in Unconsolidated Sediments

After the borehole is drilled to the desired depth, well installation can begin. The procedure for well installation will partially be dictated by the stability of the formation in which the well is being placed. If the borehole collapses immediately after the drilling tools are withdrawn, then a temporary casing must be installed and well installation will proceed through the center of the temporary casing, and continue as the temporary casing is withdrawn from the borehole. In the case of hollow-stem auger drilling, the augers will act to stabilize the borehole during well installation.

Before the screen and riser pipe are lowered into the borehole, all pipe and screen sections should be measured with an engineer's rule to ensure proper placement. When measuring sections, the threads on one end of the pipe or screen must be excluded while measuring, since the pipe and screen sections are screwed flush together.

After the screen and riser pipe are lowered through the temporary casing, the sand pack can be installed. A weighted tape measure must be used during the installation procedure to carefully monitor installation progress. The sand is slowly poured into the annulus between the riser pipe and temporary casing, as the casing is withdrawn. Sand should always be kept within the temporary casing during withdrawal in order to ensure an adequate sand pack. However, if too much sand is within the temporary casing (greater than 1 foot above the bottom of the casing) bridging between the temporary casing and riser pipe may occur. Centralizers may be used at the geologist's discretion, one above and one below the screen, to assure enough annular space for sand pack placement.

After the sand pack is installed to the desired depth (at least 1 foot above the top of the screen), then the bentonite pellet seal (or equivalent), can be installed in the same manner as the sand pack. At least 1 to 3 feet of bentonite pellets should be installed above the sand pack. Pellets should be added slowly and their fall monitored closely to ensure that bridging does not occur.

The cement-bentonite grout is then mixed and tremied into the annulus as the temporary casing or augers are withdrawn. Finally, the protective casing can be installed as detailed in Section 5.2.4.

5.3.2 Confining Layer Monitoring Wells

When drilling and installing a well in a confined aquifer, proper well installation techniques must be applied to avoid cross contamination between aquifers. Under most conditions, this can be accomplished by installing double-cased wells. This is accomplished by drilling a large-diameter boring through the upper aquifer, 1 to 5 feet into the underlying confining layer, and setting and pressure grouting or tremie grouting a large-diameter casing into the confining layer. The grout material must fill the space between the native material and the outer casing. A smaller diameter boring is then continued through the confining layer for installation of the monitoring well as detailed for overburden monitoring wells. Sufficient time (determined by the field geologist), must be allowed for setting of the grout prior to drilling through the confined layer.

5.3.3 Bedrock Monitoring Wells

When installing bedrock monitoring wells, a large diameter boring is drilled through the overburden and approximately 5 –10 feet into bedrock. A casing (typically steel) is installed and either pressure grouted or tremie grouted in place. After the grout has cured, a smaller diameter boring is continued into bedrock to the desired depth. If the boring does not collapse, the well can be left open, and a screen is not necessary. If the boring collapses, then a screen is required and can be installed as detailed for overburden monitoring wells. If a screen is to be used, then the casing which is installed through the overburden and into the bedrock does not require grouting and can be removed when the final well installation is completed.

5.3.4 Drive Points

Drive points can be installed with either a sledge hammer, drop hammer, or a mechanical vibrator. The screen section is threaded and tightened onto the riser pipe with pipe wrenches. The drive point is simply pounded into the subsurface to the desired depth. If a heavy drop hammer is used, then a tripod and pulley setup is required to lift the hammer. Drive points typically cannot be manually driven to depths exceeding 10 feet.

Direct push sampling/monitoring point installation methods, using a direct push rig or drilling rig, are described in SOP SA-2.5.

5.3.5 Innovative Monitoring Well Installation Techniques

Certain innovative sampling devices have proven advantageous. These devices are essentially screened samplers installed in a borehole with only small-diameter tubes extending to the surface. This reduces drilling costs, decreases the volume of stagnant water, and provides a sampling system that minimizes cross-contamination from sampling equipment. Four manufacturers of these samplers include Timco Manufacturing Company, Inc., of Prairie du Sac, Wisconsin, BARCAD Systems, Inc., of Concord, Massachusetts, Westbay Instruments Ltd. of Vancouver, British Columbia, Canada and the University of Waterloo at Waterloo, Ontario, Canada.. Each manufacturer offers various construction materials.

5.4 <u>Well Development Methods</u>

The purpose of well development is to stabilize and increase the permeability of the gravel pack around the well screen, and to restore the permeability of the formation which may have been reduced by drilling operations. Wells are typically developed until all fine material and drilling water is removed from the well. Sequential measurements of pH, conductivity, turbidity, and temperature taken during development may yield information (stabilized values) regarding whether sufficient development has been performed. The selection of the well development method shall be made by the field geologist and is based on the drilling methods, well construction and installation details, and the characteristics of the formation that the well is screened in. The primary methods of well development are summarized below. A more detailed discussion may be found in Driscoll (1986).

5.4.1 Overpumping and Backwashing

Wells may be developed by alternatively drawing the water level down at a high rate (by pumping or bailing) and then reversing the flow direction (backwashing) so that water is passing from the well into the formation. This back and forth movement of water through the well screen and gravel pack serves to remove fines from the formation immediately adjacent to the well, while preventing bridging (wedging) of sand grains. Backwashing can be accomplished by several methods, including pouring water into the well and then bailing, starting and stopping a pump intermittently to change water levels, or forcing water into the well under pressure through a water-tight fitting ("rawhiding"). Care should be taken when backwashing not to apply too much pressure, which could damage or destroy the well screen.

5.4.2 Surging with a Surge Plunger

A surge plunger (also called a surge block) is approximately the same diameter as the well casing and is aggressively moved up and down within the well to agitate the water, causing it to move in and out of the screens. This movement of water pulls fine materials into the well, where they may be removed by any of several methods, and prevents bridging of sand particles in the gravel pack. There are two basic types of surge plungers; solid and valved surge plungers. In formations with low yields, a valved surge plunger may be preferred, as solid plungers tend to force water out of the well at a greater rate than it will flow back in. Valved plungers are designed to produce a greater inflow than outflow of water during surging.

5.4.3 Compressed Air

Compressed air can be used to develop a well by either of two methods: backwashing or surging. Backwashing is done by forcing water out through the screens, using increasing air pressure inside a sealed well, then releasing the pressurized air to allow the water to flow back into the well. Care should be taken when using this method so that the water level does not drop below the top of the screen, thus introducing air into the formation and reducing well yield. Surging, or the "open well" method, consists of alternately releasing large volumes of air suddenly into an open well below the water level to produce a strong surge by virtue of the resistance of water head, friction, and inertia. Pumping of the well is subsequently done using the air lift method.

5.4.4 High Velocity Jetting

In the high velocity jetting method, water is forced at high velocities from a plunger-type device and through the well screen to loosen fine particles from the sand pack and surrounding formation. The jetting tool is slowly rotated and raised and lowered along the length of the well screen to develop the entire screened area. Jetting using a hose lowered into the well may also be effective. The fines washed into the screen during this process can then be bailed or pumped from the well.

6.0 RECORDS

A critical part of monitoring well installation is recording of all significant details and events in the site logbook or field notebook. The geologist must record the exact depths of significant hydrogeological features, screen placement, gravel pack placement, and bentonite placement.

A Monitoring Well Sheet (see Attachments to SOP SA-6.3) shall be completed, ensuring the uniform recording of data for each installation and rapid identification of missing information. Well depth, length, materials of construction, length and openings of screen, length and type of riser, and depth and type of all backfill materials shall be recorded. Additional information shall include location, installation date, problems encountered, water levels before and after well installation, cross-reference to the geologic boring log, and methods used during the installation and development process. Documentation is very important to prevent problems involving questionable sample validity. Somewhat different information will need to be recorded, depending on whether the well is completed in overburden (single- or double-cased), as a cased well in bedrock, or as an open hole in bedrock.

The quantities of sand, bentonite, and grout placed in the well are also important. The geologist shall calculate the annular space volume and have an idea of the quantity of material needed to fill the annular space. Volumes of backfill significantly higher than the calculated volume may indicate a problem such as a large cavity, while a smaller backfill volume may indicate a cave-in or bridging of the backfill materials. Any problems with rig operation or down-time shall be recorded and may affect the driller's final fee.

7.0 REFERENCES

Scalf, M. R., J. F. McNabb, W. J. Dunlap, R. L. Cosby, and J. Fryberger, 1981. Manual of Groundwater Sampling Procedures. R. S. Kerr Environmental Research Laboratory, Office of Research and Development, U.S. EPA, Ada, Oklahoma.

Barcelona, M. J., P. P. Gibb and R. A. Miller, 1983. A Guide to the selection of Materials for Monitoring Well Construction and Groundwater Sampling. ISWS Contract Report 327, Illinois State Water Survey, Champaign, Illinois.

U.S. EPA, 1980. Procedures Manual for Groundwater Monitoring of Solid Waste Disposal Facilities. Publication SW-611, Office of Solid Waste, U.S. EPA, Washington, D.C.

Driscoll, Fletcher G., 1986. Groundwater and Wells. Johnson Division, St. Paul, Minnesota, 1989.

ATTACHMENT A

RELATIVE COMPATIBILITY OF RIGID WELL CASING MATERIAL (PERCENT)

Potentially-Deteriorating Substance	Type of Casing Material						
	PVC 1	Galvanized Steel	Carbon Steel	Lo-carbon Steel	Stainless Steel 304	Stainless Steel 316	Teflon*
Buffered Weak Acid	100	56	51	59	97	100	100
Weak Acid	98	59	43	47	96	100	100
Mineral Acid/ High Solids Content	100	48	57	60	80	82	100
Aqueous/Organic Mixtures	64	69	73	73	98	100	100
Percent Overall Rating	91	58	56	59	93	96	100

<u>Preliminary Ranking of Rigid Materials</u>:

1 Teflon®
2 Stainless Steel 316
3. Stainless Steel 304
4 PVC 1
5 Lo-Carbon Steel
6 Galvanized Steel
7 Carbon Steel

* Trademark of DuPont

RELATIVE COMPATIBILITY OF SEMI-RIGID OR ELASTOMERIC MATERIALS (PERCENT)

Potentially-Deteriorating Substance	Type of Casing Material							
	PVC Flexible	PP	PE Conv.	PE Linear	PMM	Viton®*	Silicone	Neoprene	Teflon®*
Buffered Weak Acid	97	97	100	97	90	92	87	85	100
Weak Acid	92	90	94	96	78	78	75	75	100
Mineral Acid/ High Solids Content	100	100	100	100	95	100	78	82	100
Aqueous/Organic Mixtures	62	71	40	60	49	78	49	44	100
Percent Overall Rating	88	90	84	88	78	87	72	72	100

<u>Preliminary Ranking of Semi-Rigid or Elastomeric Materials</u>:

1 Teflon®
2 Polypropylene (PP)
3. PVC Flexible/PE Linear
4 Viton®
5 PE Conventional
6 Plexiglas/Lucite (PMM)
7 Silicone/Neoprene

* Trademark of DuPont

Source: Barcelona et al., 1983

ATTACHMENT B

COMPARISON OF STAINLESS STEEL AND PVC FOR MONITORING WELL CONSTRUCTION

Characteristic	Stainless Steel	PVC
Strength	Use in deep wells to prevent compression and closing of screen/riser.	Use when shear and compressive strength are not critical.
Weight	Relatively heavier.	Light-weight; floats in water.
Cost	Relatively expensive.	Relatively inexpensive.
Corrosivity	Deteriorates more rapidly in corrosive water.	Non-corrosive -- may deteriorate in presence of ketones, aromatics, alkyl sulfides, or some chlorinated hydrocarbons.
Ease of Use	Difficult to adjust size or length in the field.	Easy to handle and work with in the field.
Preparation for Use	Should be steam cleaned if organics will be subsequently sampled.	Never use glue fittings -- pipes should be threaded or pressure fitted. Should be steam cleaned when used for monitoring wells.
Interaction with Contaminants*	May sorb organic or inorganic substances when oxidized.	May sorb or release organic substances.

* See also Attachment A.

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		Effective Date 12/03	Revision 2
		Applicability Tetra Tech NUS, Inc.	
		Prepared Health & Safety	
Subject UTILITY LOCATING AND EXCAVATION CLEARANCE		Approved D. Senovich	

TABLE OF CONTENTS

1.0 PURPOSE

Utilities such as electric service lines, natural or propane gas lines, water and sewage lines, telecommunications, and steam lines are very often in the immediate vicinity of work locations. Contact with underground or overhead utilities can have serious consequences including employee injury/fatality, property and equipment damage, substantial financial impacts, and loss of utility service to users.

The purpose of this procedure is to provide minimum requirements and technical guidelines regarding the appropriate procedures to be followed when performing subsurface and overhead utility locating services. It is the policy of Tetra Tech NUS, Inc. (TtNUS) to provide a safe and healthful work environment for the protection of our employees. The purpose of this Standard Operating Procedure (SOP) is to aid in achieving the objectives of this policy, to present the acceptable procedures pertaining to utility locating and excavation clearance activities, and to present requirements and restrictions relevant to these types of activities. This SOP must be reviewed by any employee potentially involved with underground or overhead utility locating and avoidance activities.

2.0 SCOPE

This procedure applies to all TtNUS field activities where there may be potential contact with underground or overhead utilities. This procedure provides a description of the principles of operation, instrumentation, applicability, and implementability of typical methods used to determine the presence and avoidance of contact with utility services. This procedure is intended to assist with work planning and scheduling, resource planning, field implementation, and subcontractor procurement. Utility locating and excavation clearance requires site-specific information prior to the initiation of any such activities on a specific project. This SOP is not intended to provide a detailed description of methodology and instrument operation. Specialized expertise during both planning and execution of several of the methods presented may also be required.

3.0 GLOSSARY

Electromagnetic Induction (EMI) Survey - A geophysical exploration method whereby electromagnetic fields are induced in the ground and the resultant secondary electromagnetic fields are detected as a measure of ground conductivity.

Magnetometer – A device used for precise and sensitive measurements of magnetic fields.

Magnetic Survey – A geophysical survey method that depends on detection of magnetic anomalies caused by the presence of buried ferromagnetic objects.

Metal Detection – A geophysical survey method that is based on electromagnetic coupling caused by underground conductive objects.

Vertical Gradiometer – A magnetometer equipped with two sensors that are vertically separated by a fixed distance. It is best suited to map near surface features and is less susceptible to deep geologic features.

Ground Penetrating Radar – Ground Penetrating Radar (GPR) involves specialized radar equipment whereby a signal is sent into the ground via a transmitter. Some portion of the signal will be reflected from the subsurface material, which is then recorded with a receiver and electronically converted into a graphic picture.

4.0 RESPONSIBILITIES

Project Manager (PM)/Task Order Manager (TOM) - Responsible for ensuring that all field activities are conducted in accordance with this procedure.

Site Manager (SM)/Field Operations Leader (FOL) - Responsible for the onsite verification that all field activities are performed in compliance with approved SOPs or as otherwise directed by the approved project plan(s).

Site Health & Safety Officer (SHSO) – Responsible to provide technical assistance and verify full compliance with this SOP. The SHSO is also responsible for reporting any deficiencies to the Corporate Health and Safety Manager (HSM) and to the PM/TOM.

Health & Safety Manager (HSM) – Responsible for preparing, implementing, and modifying corporate health and safety policy and this SOP.

Site Personnel – Responsible for performing their work activities in accordance with this SOP and the TtNUS Health and Safety Policy.

5.0 PROCEDURES

This procedure addresses the requirements and technical procedures that must be performed to minimize the potential for contact with underground and overhead utility services. These procedures are addressed individually from a buried and overhead standpoint.

5.1 Buried Utilities

Buried utilities present a heightened concern because their location is not typically obvious by visual observation, and it is common that their presence and/or location is unknown or incorrectly known on client properties. This procedure must be followed prior to beginning any subsurface probing or excavation that might potentially be in the vicinity of underground utility services. In addition, the Utility Clearance Form (Attachment 3) must be completed for every location or cluster of locations where intrusive activities will occur.

Where the positive identification and de-energizing of underground utilities cannot be obtained and confirmed using the following steps, the PM/TOM is responsible for arranging for the procurement of a qualified, experienced, utility locating subcontractor who will accomplish the utility location and demarcation duties specified herein.

1. A comprehensive review must be made of any available property maps, blue lines, or as-builts prior to site activities. Interviews with local personnel familiar with the area should be performed to provide additional information concerning the location of potential underground utilities. Information regarding utility locations shall be added to project maps upon completion of this exercise.

2., A visual site inspection must be performed to compare the site plan information to actual field conditions. Any findings must be documented and the site plan/maps revised. The area(s) of proposed excavation or other subsurface activities must be marked at the site in white paint or pin flags to identify those locations of the proposed intrusive activities. The site inspection should focus on locating surface indications of potential underground utilities. Items of interest include the presence of nearby area lights, telephone service, drainage grates, fire hydrants, electrical service vaults/panels, asphalt/concrete scares and patches, and topographical depressions. Note the location of any emergency shut off switches. Any additional information regarding utility locations shall be added to project maps upon completion of this exercise and returned to the PM/TOM.

3. If the planned work is to be conducted on private property (e.g., military installations, manufacturing facilities, etc.) the FOL must identify and contact appropriate facility personnel (e.g., public works or facility engineering) before any intrusive work begins to inquire about (and comply with) property owner requirements. It is important to note that private property owners may require several days to several weeks advance notice prior to locating utilities.

4. If the work location is on public property, the state agency that performs utility clearances must be notified (see Attachment 1). State "one-call" services must be notified prior to commencing fieldwork per their requirements. Most one-call services require, by law, 48- to 72-hour advance notice prior to beginning any excavation. Such services typically assign a "ticket" number to the particular site. This ticket number must be recorded for future reference and is valid for a specific period of time, but may be extended by contacting the service again. The utility service will notify utility representatives who then mark their respective lines within the specified time frame. It should be noted that most military installations own their own utilities but may lease service and maintenance from area providers. Given this situation, "one call" systems may still be required to provide location services on military installations.

5. Utilities must be identified and their locations plainly marked using pin flags, spray paint, or other accepted means. The location of all utilities must be noted on a field sketch for future inclusion on project maps. Utility locations are to be identified using the following industry-standard color code scheme, unless the property owner or utility locator service uses a different color code:

white	excavation/subsurface investigation location
red	electrical
yellow	gas, oil, steam
orange	telephone, communications
blue	water, irrigation, slurry
green	sewer, drain

6. Where utility locations are not confirmed with a high degree of confidence through drawings, schematics, location services, etc., the work area must be thoroughly investigated prior to beginning the excavation. In these situations, utilities must be identified using safe and effective methods such as passive and intrusive surveys, or the use of non-conductive hand tools. Also, in situations where such hand tools are used, they should always be used in conjunction with suitable detection equipment, such as the items described in Section 6.0 of this SOP. Each method has advantages and disadvantages including complexity, applicability, and price. It also should be noted that in some states, initial excavation is required by hand to a specified depth.

7. At each location where trenching or excavating will occur using a backhoe or other heavy equipment, and where utility identifications and locations cannot be confirmed prior to groundbreaking, the soil must be probed using a device such as a tile probe which is made of non-conductive material such as fiberglass. If these efforts are not successful in clearing the excavation area of suspect utilities, hand shoveling must be performed for the perimeter of the intended excavation.

8. All utilities uncovered or undermined during excavation must be structurally supported to prevent potential damage. Unless necessary as an emergency corrective measure, TtNUS shall not make any repairs or modifications to existing utility lines without prior permission of the utility owner, property owner, and Corporate HSM. All repairs require that the line be locked-out/tagged-out prior to work.

5.2 Overhead Power Lines

If it is necessary to work within the minimum clearance distance of an overhead power line, the overhead line must be de-energized and grounded, or re-routed by the utility company or a registered electrician. If protective measures such as guarding, isolating, or insulating are provided, these precautions must be adequate to prevent employees from contacting such lines directly with any part of their body or indirectly though conductive materials, tools, or equipment.

The following table provides the required minimum clearances for working in proximity to overhead power lines.

Nominal Voltage	Minimum Clearance
0 -50 kV	10 feet, or one mast length; whichever is greater
50+ kV	10 feet plus 4 inches for every 10 kV over 50 kV or 1.5 mast lengths; whichever is greater

6.0 UNDERGROUND LOCATING TECHNIQUES

A variety of supplemental utility locating approaches are available and can be applied when additional assurance is needed. The selection of the appropriate method(s) to employ is site-specific and should be tailored to the anticipated conditions, site and project constraints, and personnel capabilities.

6.1 Geophysical Methods

Geophysical methods include electromagnetic induction, magnetics, and ground penetrating radar. Additional details concerning the design and implementation of electromagnetic induction, magnetics, and ground penetrating radar surveys can be found in one or more of the TtNUS SOPs included in the References (Section 8.0).

Electromagnetic Induction

Electromagnetic Induction (EMI) line locators operate either by locating a background signal or by locating a signal introduced into the utility line using a transmitter. A utility line acts like a radio antenna, producing electrons, which can be picked up with a radiofrequency receiver. Electrical current carrying conductors have a 60HZ signal associated with them. This signal occurs in all power lines regardless of voltage. Utilities in close proximity to power lines or used as grounds may also have a 60HZ signal, which can be picked up with an EM receiver. A typical example of this type of geophysical equipment is an EM-61.

EMI locators specifically designed for utility locating use a special signal that is either indirectly induced onto a utility line by placing the transmitter above the line or directly induced using an induction clamp. The clamp induces a signal on the specific utility and is the preferred method of tracing since there is little chance of the resulting signals being interfered with. A good example of this type of equipment is the Schonstedt® MAC-51B locator. The MAC-51B performs inductively traced surveys, simple magnetic locating, and traced nonmetallic surveys.

When access can be gained inside a conduit to be traced, a flexible insulated trace wire can be used. This is very useful for non-metallic conduits but is limited by the availability of gaining access inside the pipe.

Magnetics

Magnetic locators operate by detecting the relative amounts of buried ferrous metal. They are incapable of locating or identifying nonferrous utility lines but can be very useful for locating underground storage tanks (UST's), steel utility lines, and buried electrical lines. A typical example of this type of equipment is the Schonstedt® GA-52Cx locator. The GA-52Cx is capable of locating 4-inch steel pipe up to 8 feet deep.

Non-ferrous lines are often located by using a typical plumbing tool (snake) fed through the line. A signal is then introduced to the snake that is then traced.

Ground Penetrating Radar

Ground Penetrating Radar (GPR) involves specialized radar equipment whereby a signal is sent into the ground via a transmitter. Some portion of the signal will be reflected from the subsurface material, which is then recorded with a receiver and electronically converted into a graphic picture. In general, an object which is harder than the surrounding soil will reflect a stronger signal. Utilities, tunnels, UST's, and footings will reflect a stronger signal than the surrounding soil. Although this surface detection method may determine the location of a utility, this method does not specifically identify utilities (i.e., water vs. gas, electrical vs. telephone); hence, verification may be necessary using other methods. This method is somewhat limited when used in areas with clay soil types or with a high water table.

6.2 Passive Detection Surveys

Acoustic Surveys

Acoustic location methods are generally most applicable to waterlines or gas lines. A highly sensitive Acoustic Receiver listens for background sounds of water flowing (at joints, leaks, etc.) or to sounds introduced into the water main using a transducer. Acoustics may also be applicable to determine the location of plastic gas lines.

Thermal Imaging

Thermal (i.e., infrared) imaging is a passive method for detecting the heat emitted by an object. Electronics in the infrared camera convert subtle heat differentials into a visual image on the viewfinder or a monitor. The operator does not look for an exact temperature; rather they look for heat anomalies (either elevated or suppressed temperatures) characteristic of a potential utility line.

The thermal fingerprint of underground utilities results from differences in temperature between the atmosphere and the fluid present in a pipe or the heat generated by electrical resistance. In addition, infrared scanners may be capable of detecting differences in the compaction, temperature and moisture content of underground utility trenches. High-performance thermal imagery can detect temperature differences to hundredths of a degree.

6.3 Intrusive Detection Surveys

Vacuum Excavation

Vacuum excavation is used to physically expose utility services. The process involves removing the surface material over approximately a 1' x 1' area at the site location. The air-vacuum process proceeds with the simultaneous action of compressed air-jets to loosen soil and vacuum extraction of the resulting debris. This process ensures the integrity of the utility line during the excavation process, as no hammers, blades, or heavy mechanical equipment comes into contact with the utility line, eliminating the risk of damage to utilities. The process continues until the utility is uncovered. Vacuum excavation can be used at the proposed site location to excavate below the "utility window" which is usually 8 feet.

Hand Excavation

When the identification and location of underground utilities cannot be positively confirmed through document reviews and/or other methods, borings and excavations may be cleared via the use of non-conductive hand tools. This should always be done in conjunction with the use of detection equipment. This would be required for all locations where there is a potential to impact buried utilities. The minimum hand-excavation depth that must be reached is to be determined considering the geographical location of the work site. This approach recognizes that the placement of buried utilities is influenced by frost line depths that vary by geographical region. Attachment 2 presents frost line depths for the regions of the contiguous United States. At a minimum, hand excavation depths must be at least to the frost line depth (see Attachment 2) plus two (2) feet, but never less than 4 feet below ground surface (bgs). For hand excavation, the hole created must be reamed large enough to be at least the diameter of the drill rig auger or bit prior to drilling. For soil gas surveys, the survey probe shall be placed as close as possible to the cleared hand excavation. It is important to note that a post-hole digger must not be used in this type of hand excavation activity.

Tile Probe Surveys

For some soil types, site conditions, and excavation requirements, non-conductive tile probes may be used. A tile probe is a "T"-handled rod of varying lengths that can be pushed into the soil to determine if any obstructions exist at that location. Tile probes constructed of fiberglass or other nonconductive material are readily-available from numerous vendors. Tile probes must be performed to the same depth requirements as previously specified. As with other types of hand excavating activities, the use of a non-conductive tile probe, should always be in conjunction with suitable utility locating detection equipment.

7.0 INTRUSIVE ACTIVITIES SUMMARY

The following list summarizes the activities that must be performed prior to beginning subsurface activities:

1. Map and mark all subsurface locations and excavation boundaries using white paint or markers specified by the client or property owner.

2. Notify the property owner and/or client that the locations are marked. At this point, drawings of locations or excavation boundaries shall be provided to the property owner and/or client so they may initiate (if applicable) utility clearance.

   Note: Drawings with confirmed locations should be provided to the property owner and/or client as soon as possible to reduce potential time delays.

3. Notify "One Call" service. If possible, arrange for an appointment to show the One Call representative the surface locations or excavation boundaries in person. This will provide a better location designation to the utilities they represent. You should have additional drawings should you need to provide plot plans to the One Call service.

4. Implement supplemental utility detection techniques as necessary and appropriate to conform utility locations or the absence thereof.

5. Complete Attachment 3, Utility Clearance Form. This form should be completed for each excavation location. In situations where multiple subsurface locations exist within the close proximity of one another, one form may be used for multiple locations provided those locations are noted on the Utility Clearance Form. Upon completion, the Utility Clearance Form and revised/annotated utility location map becomes part of the project file.

8.0 REFERENCES

OSHA Letter of Interpretation, Mr. Joseph Caldwell, Attachment 4
OSHA 29 CFR 1926(b)(2)
OSHA 29 CFR 1926(b)(3)
TtNUS Utility Locating and Clearance Policy
TtNUS SOP GH-3.1; Resistivity and Electromagnetic Induction
TtNUS SOP GH-3.2; Magnetic and Metal Detection Surveys
TtNUS SOP GH-3.4; Ground-penetrating Radar Surveys

ATTACHMENT 1
LISTING OF UNDERGROUND UTILITY CLEARANCE RESOURCES

American Public Works Association
2345 Grand Boulevard, Suite 500, Kansas City, MO 64108-2625
Phone (816) 472-6100 • Fax (816) 472-1610
Web www.apwa.net • E-mail apwa@apwa.net

ONE-CALL SYSTEMS INTERNATIONAL
CONDENSED DIRECTORY

Alabama
Alabama One-Call
1-800-292-8525

Alaska
Locate Call Center of Alaska, Inc.
1-800-478-3121

Arizona
Arizona Blue Stake
1-800-782-5348

Arkansas
Arkansas One Call System, Inc.
1-800-482-8998

California
Underground Service Alert North
1-800-227-2600
Underground Service Alert of Southern California
1-800-227-2600

Colorado
Utility Notification Center of Colorado
1-800-922-1987

Connecticut
Call Before You Dig
1-800-922-4455

Delaware
Miss Utility of Delmarva
1-800-282-8555

Florida
Sunshine State One-Call of Florida, Inc.
1-800-432-4770

Georgia
Underground Protection Center, Inc.
1-800-282-7411

Hawaii
Underground Service Alert North
1-800-227-2600

Idaho
Dig Line Inc.
1-800-342-1585
Kootenai County One-Call
1-800-428-4950
Shoshone - Benewah One-Call
1-800-398-3285

Illinois
JULIE, Inc.
1-800-892-0123
Digger (Chicago Utility Alert Network)
312-744-7000

Indiana
Indiana Underground Plant Protection Service
1-800-382-5544

Iowa
Iowa One-Call
1-800-292-8989

Kansas
Kansas One-Call System, Inc.
1-800-344-7233

Kentucky
Kentucky Underground Protection Inc.
1-800-752-6007

Louisiana
Louisiana One Call System, Inc.
1-800-272-3020

Maine
Dig Safe System, Inc.
1-888-344-7233

Maryland
Miss Utility
1-800-257-7777
Miss Utility of Delmarva
1-800-282-8555

Massachusetts
Dig Safe System, Inc.
1-888-344-7233

Michigan
Miss Dig System, Inc.
1-800-482-7171

Minnesota
Gopher State One Call
1-800-252-1166

Mississippi
Mississippi One-Call System, Inc
1-800-227-6477

Missouri
Missouri One-Call System, Inc.
1-800-344-7483

Montana
Utilities Underground Protection Center
1-800-424-5555
Montana One Call Center
1-800-551-8344

Nebraska
Diggers Hotline of Nebraska
1-800-331-5666

Nevada
Underground Service Alert North
1-800-227-2600

New Hampshire
Dig Safe System, Inc.
1-888-344-7233

New Jersey
New Jersey One Call
1-800-272-1000

New Mexico
New Mexico One Call System, Inc.
1-800-321-2537
Las Cruces- Dona Ana Blue Stakes
1-888-526-0400

New York
Dig Safely New York
1-800-962-7962
New York City- Long Island One Call Center
1-800-272-4480

North Carolina
The North Carolina One-Call Center, Inc.
1-800-632-4949

North Dakota
North Dakota One-Call
1-800-795-0555

Ohio
Ohio Utilities Protection Service
1-800-362-2764
Oil & Gas Producers Underground Protect'n Svc
1-800-925-0988

Oklahoma
Call Okie
1-800-522-6543

Oregon
Oregon Utility Notification Center/One Call Concepts
1-800-332-2344

Pennsylvania
Pennsylvania One Call System, Inc.
1-800-242-1776

Rhode Island
Dig Safe System, Inc.
1-888-344-7233

South Carolina
Palmetto Utility Protection Service Inc.
1-888-721-7877

South Dakota
South Dakota One Call
1-800-781-7474

Tennessee
Tennessee One-Call System, Inc.
1-800-351-1111

ATTACHMENT 1 (Continued)

Texas
Texas One Call System
1-800-245-4545
Texas Excavation Safety System, Inc.
1-800-344-8377
Lone Star Notification Center
1-800-669-8344

Utah
Blue Stakes of Utah
1-800-662-4111

Vermont
Dig Safe System, Inc.
1-888-344-7233

Virginia
Miss Utility of Virginia
1-800-552-7001
Miss Utility (Northern Virginia)
1-800-257-7777

Washington
Utilities Underground Location Center
1-800-424-5555
Northwest Utility Notification Center
1-800-553-4344
Inland Empire Utility Coordinating Council
509-456-8000

West Virginia
Miss Utility of West Virginia, Inc.
1-800-245-4848

Wisconsin
Diggers Hotline, Inc.
1-800-242-8511

Wyoming
Wyoming One-Call System, Inc.
1-800-348-1030
Call Before You Dig of Wyoming
1-800-849-2476

District of Columbia
Miss Utility
1-800-257-7777

Alberta
Alberta One-Call Corporation
1-800-242-3447

British Columbia
BC One Call
1-800-474-6886

Ontario
Ontario One-Call System
1-800-400-2255

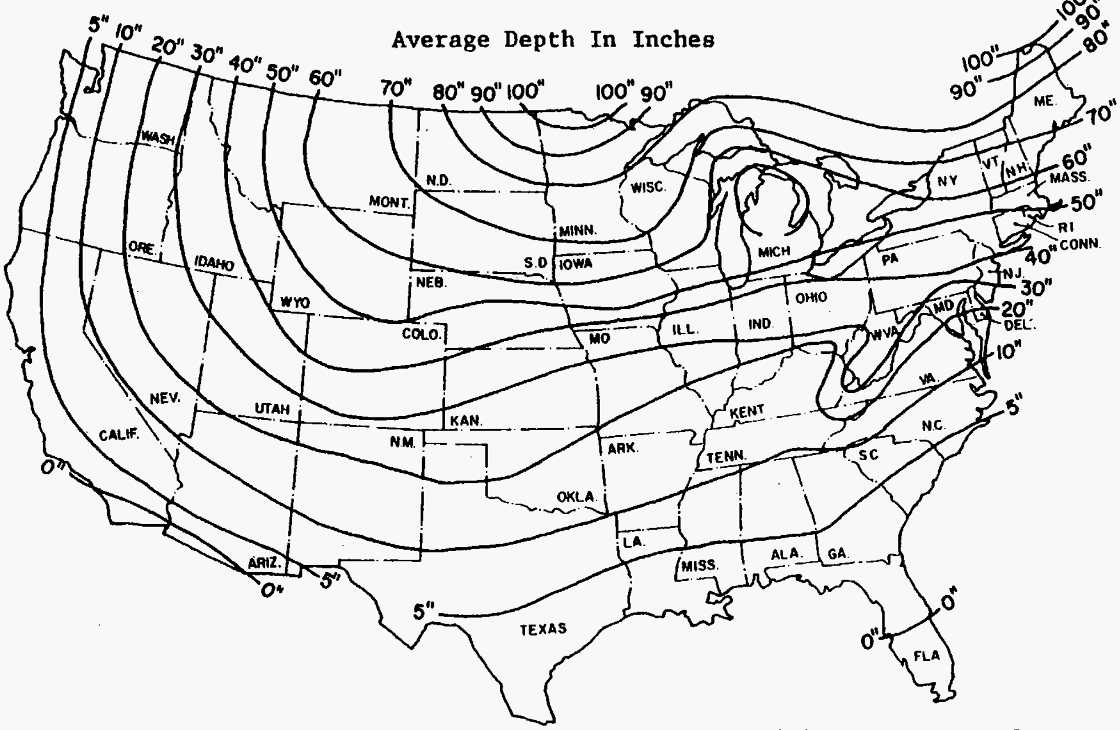
Quebec
Info-Excavation
1-800-663-9228

ATTACHMENT 2

FROST LINE PENETRATION DEPTHS BY GEOGRAPHIC LOCATION

FROST PENETRATION

Average Depth In Inches



Courtesy U.S. Department Of Commerce

ATTACHMENT 3
UTILITY CLEARANCE FORM

Client: ____________________ Project Name: ________________________________

Project No.: ______________________ Completed By: ________________________________

Location Name: __________________________ Work Date: ____________________________

Excavation Method/Overhead Equipment: ____

1. Underground Utilities — Circle One

a)	Review of existing maps?	yes no N/A
b)	Interview local personnel?	yes no N/A
c)	Site visit and inspection?	yes no N/A
d)	Excavation areas marked in the field?	yes no N/A
e)	Utilities located in the field?	yes no N/A
f)	Located utilities marked/added to site maps?	yes no N/A
g)	Client contact notified Name ______________ Telephone: _________ Date: _____________	yes no N/A
g)	State One-Call agency called? Caller: ______________________________ Ticket Number: _________________________ Date: _____________	yes no N/A
h)	Geophysical survey performed? Survey performed by: ___________________ Method: ______________________________ Date: _____________	yes no N/A
i)	Hand excavation performed (with concurrent use of utility detection device)? Completed by: _________________________ Total depth: __________feet Date: _______	yes no N/A
j)	Trench/excavation probed? Probing completed by: __________________ Depth/frequency: _______________________ Date: _____________	yes no N/A

2. Overhead Utilities — Present Absent

a)	Determination of nominal voltage	yes no N/A
b)	Marked on site maps	yes no N/A
c)	Necessary to lockout/insulate/re-route	yes no N/A
d)	Document procedures used to lockout/insulate/re-route	yes no N/A
e)	Minimum acceptable clearance (SOP Section 5.2): _________________	

3. Notes:

____

____

____

Approval:

_________________________________ _________________

Site Manager/Field Operations Leader Date

c: PM/Project File
Program File

ATTACHMENT 4
OSHA LETTER OF INTERPRETATION

Mr. Joseph Caldwell
Consultant
Governmental Liaison
Pipeline Safety Regulations
211 Wilson Boulevard
Suite 700
Arlington, Virginia 22201

Re: Use of hydro-vacuum or non-conductive hand tools to locate underground utilities.

Dear Mr. Caldwell:

In a letter dated July 7, 2003, we responded to your inquiry of September 18, 2002, regarding the use of hydro-vacuum equipment to locate underground utilities by excavation. After our letter to you was posted on the OSHA website, we received numerous inquiries that make it apparent that aspects of our July 7 letter are being misunderstood. In addition, a number of industry stakeholders, including the National Utility Contractors Association (NUCA), have provided new information regarding equipment that is available for this work.

To clarify these issues, we are withdrawing our July 7 letter and issuing this replacement response to your inquiry.

Question: *Section 1926.651 contains several requirements that relate to the safety of employees engaged in excavation work. Specifically, paragraphs (b)(2) and (b)(3) relate in part to the safety of the means used to locate underground utility installations that, if damaged during an uncovering operation, could pose serious hazards to employees.*

Under these provisions, what constitutes an acceptable method of uncovering underground utility lines, and further, would the use of hydro-vacuum excavation be acceptable under the standard?

Answer

Background

Two sections of 29 CFR 1926 Subpart P (Excavations), 1926.651(Specific excavation requirements), govern methods for uncovering underground utility installations. Specifically, paragraph (b)(2) states:

> When utility companies or owners cannot respond to a request to locate underground utility installations within 24 hours * * * or cannot establish the exact location of these installations, the employer may proceed, provided the employer does so with caution, and provided detection equipment or other acceptable means to locate utility installations are used. (emphasis added).

Paragraph (b)(3) provides:

ATTACHMENT 4 (Continued)

> When excavation operations approach the estimated location of underground installations, the exact location of the installations shall be determined by safe and acceptable means. (emphasis added).

Therefore, "acceptable means" must be used where the location of the underground utilities have not been identified by the utility companies and detection equipment is not used.

Subpart P does not contain a definition of either "other acceptable means" or "safe and acceptable means." The preambles to both the proposed rule and the final rule discussed the rationale behind the wording at issue. For example, the preamble to the proposed rule, 52 Fed. Reg. 12301 (April 15, 1987), noted that a 1972 version of this standard contained language that specified "careful probing or hand digging" as the means to uncover utilities. The preamble then noted that an amendment to the 1972 standard later deleted that language "to allow other, *equally effective means* of locating such installations." The preamble continued that in the 1987 proposed rule, OSHA again proposed using language in section (b)(3) that would provide another example of an acceptable method of uncovering utilities that could be used *where the utilities have not been marked and detection equipment is not being used* – "probing with hand-held tools." This method was rejected in the final version of 29 CFR 1926. As OSHA explained in the preamble to the final rule, 54 Fed. Reg. 45916 (October 31, 1989):

> OSHA received two comments * * * and input from ACCSH [OSHA's Advisory Committee on Construction Safety and Health] * * * on this provision. All commenters recommended dropping 'such as probing with hand-held tools' from the proposed provision, because this could create a hazard to employees by damaging the installation or its insulation.

In other words, the commenters objected to the use of hand tools being used unless detection equipment was used in conjunction with them. OSHA then concluded its discussion relative to this provision by agreeing with the commentators and ultimately not including any examples of "acceptable means" in the final provision.

Non-conductive hand tools are permitted

This raises the question of whether the standard permits the use of hand tools alone -- without also using detection equipment. NUCA and other industry stakeholders have recently informed us that non-conductive hand tools that are appropriate to be used to locate underground utilities are now commonly available.

Such tools, such as a "shooter" (which has a non-conductive handle and a snub nose) and non-conductive or insulated probes were not discussed in the rulemaking. Since they were not considered at that time, they were not part of the class of equipment that was thought to be unsafe for this purpose. Therefore, we conclude that the use of these types of hand tools, when used with appropriate caution, is an "acceptable means" for locating underground utilities.

ATTACHMENT 4 (Continued)

Hydro-vacuum excavation

It is our understanding that some hydro-vacuum excavation equipment can be adjusted to use a minimum amount of water and suction pressure. When appropriately adjusted so that the equipment will not damage underground utilities (especially utilities that are particularly vulnerable to damage, such as electrical lines), use of such equipment would be considered a "acceptable means" of locating underground utilities. However, if the equipment cannot be sufficiently adjusted, then this method would not be acceptable under the standard.

Other technologies

We are not suggesting that these are the only devices that would be "acceptable means" under the standard. Industry stakeholders have informed us that there are other types of special excavation equipment designed for safely locating utilities as well.

We apologize for any confusion our July 7 letter may have caused. If you have further concerns or questions, please feel free to contact us again by fax at: U.S. Department of Labor, OSHA, Directorate of Construction, Office of Construction Standards and Compliance Assistance, fax # 202-693-1689. You can also contact us by mail at the above office, Room N3468, 200 Constitution Avenue, N.W., Washington, D.C. 20210, although there will be a delay in our receiving correspondence by mail.

Sincerely,

Russell B. Swanson, Director
Directorate of Construction

NOTE: OSHA requirements are set by statute, standards and regulations. Our interpretation letters explain these requirements and how they apply to particular circumstances, but they cannot create additional employer obligations. This letter constitutes OSHA=s interpretation of the requirements discussed. Note that our enforcement guidance may be affected by changes to OSHA rules. Also, from time to time we update our guidance in response to new information. To keep apprised of such developments, you can consult OSHA's website at http://www.osha.gov.

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		Effective Date 09/03	Revision 2
		Applicability Tetra Tech NUS, Inc.	
		Prepared Earth Sciences Department	
Subject SOIL GAS SAMPLING		Approved D. Senovich	

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1.0 PURPOSE

The purpose of this procedure is to provide general reference information on soil gas sampling. Soil gas investigations measure the general extent of volatile organic compound (VOC) contamination, such as chlorinated solvents and petroleum products, given off by subsurface soil and groundwater. The methods and equipment described are for collection of soil gas from the unsaturated zone of the subsurface soil.

2.0 SCOPE

This procedure provides information on proper sampling equipment and techniques for soil gas sampling. Review of the information contained herein will facilitate planning of the field sampling effort by describing standard sampling techniques. The techniques described shall be followed whenever applicable, noting that site-specific conditions or project-specific plans may require adjustments in methodology.

3.0 GLOSSARY

<u>Sampling Grid</u> - Typically consists of a series of equal-distant sampling points set along parallel survey lines. The sample point spacing and number of parallel grid lines will depend upon the site specific conditions and project objectives.

<u>Transect Line</u> - A sampling network used to find the source area of contamination. Sampling points are placed along a transect line between the area of impact and a suspected source area. This can significantly decrease the number of points compared to a typical sampling grid.

<u>Biased Location</u> - Sample points are either placed near a suspected source area or in an anticipated clean area to refine the location of "hot spots" for further delineation or remediation.

<u>Random Location</u> - Random networks use a grid with numbers designating the nodes or areas within the grid. The sample points are then selected by a random number generator to designate which nodes or areas are targeted for sampling. This type of network is used in areas where little information is known or no contamination is suspected.

<u>Combined Locations</u> - This type of network is the most common used, and includes a combination of any of the four previous sampling networks mentioned.

<u>Head Space Analysis</u> - The screening or analysis of volatile organic vapors that have accumulated in the air space within a soil or groundwater sample container.

<u>Flame Ionization Detector (FID)</u> - A portable instrument for the measurement of many combustible organic compounds and a few inorganic compounds in air at parts-per million (ppm) levels. The basis for the detection is the ionization of gaseous species utilizing a flame as the energizing source.

<u>Photo Ionization Detector (PID)</u> - A portable instrument for the measurement of many combustible organic compounds and a few inorganic compounds in air at ppm levels. A PID will not detect methane gas. The basis for the detection is the ionization of gaseous species utilizing ultraviolet radiation as the energizing source.

<u>Direct Push Technology (DPT)</u> - DPT refers to sampling tools and sensors that are driven directly into the ground without the use of conventional drilling equipment. DPT typically utilizes hydraulic pressure and/or percussion hammers to advance the sampling tools. A primary advantage of DPT over conventional drilling techniques is that DPT results in the generation of little or no investigation derived waste (soil cuttings, purge water, etc.).

4.0 RESPONSIBILITIES

Project Manager - The Project Manager is responsible for selecting and/or reviewing the appropriate soil gas sampling procedure required to support the project objectives.

Field Operations Leader (FOL)-The FOL is primarily responsible for performing the soil gas sampling technique in accordance with the project-specific plan.

5.0 PROCEDURES

5.1 General

Soil gas methods are performed to delineate VOC contamination in subsurface soil and groundwater. Soil gas surveys are not intended to be a substitute for conventional subsurface methodologies (e.g., monitoring well installation and groundwater sampling) but rather are to be used as a screening technique to focus subsequent investigations to areas of potential concern. The advantages of using soil gas methodologies to define VOC contamination include:

1. Minimizing the number of subsequent test borings and monitoring wells required to characterize the nature and extent of the contamination.
2. Optimizing the placement of subsequent monitoring wells and test borings.
3. Allowing the collection of large amounts of data in a short time period relative to conventional methods.
4. Generating little or no investigative derived waste.

To assess the effectiveness of a soil gas survey, consider the following items:

1. The near surface geology.
2. The type of contamination present.
3. The anticipated concentration of contamination present.
4. The anticipated depth to the zone of contamination.

Common soil gas methods are most effective in geological settings that contain coarse textured soils (e.g., silty sands, sands and gravels) and are least effective in areas of fine textured soils (e.g., silty clays and clays). Soil gas methods may be effective at mapping areas of dense non-aqueous phase liquid VOC contamination that are present beneath the water table. Areas with deep water tables or low concentrations of VOC contamination may require the use of specialized soil gas techniques. These techniques could require the use of more expensive passive sorbent samplers left in place for a relatively long time period.

5.2 Soil Gas Sampling Grids

The first stage of a soil gas survey is to establish a sampling grid or network. The grid should be designed to obtain all necessary information with a minimal expenditure of time and resources. The development of the grid should be based on background information regarding chemical properties of the contaminant, properties of the vadose zone, and hydrogeologic conditions of the area. All of this information should be used to design a sampling protocol specific to the conditions at the site. Some of the designs used include grids, transect lines, and biased, random or combined methodologies.

The size of the grid spacing is determined on a site specific basis. Locations should be marked with pin flags or wooden stakes and numbered sequentially at each site. The grid should be referenced to permanent site features and the stakes left in place to assist with future subsurface investigations. The location of any nearby surface or subsurface features which may affect the results of the survey (sewer line, fuel farm, etc.) shall be noted in the field notebook. Detailed notes and a sketch of each site, including station locations and station numbers will be documented in the field logbook. After the grid has been laid-out a dig permit must be applied for and approved prior to the start of sampling.

5.3 Sampling Methodologies

There are several methods for the collection of soil gas samples. The most common types will be discussed in the following sections. Variations of the following methods may be conducted if approved by the Project Manager.

5.3.1 Head Space Screening

Head space screening is a method of obtaining rapid information concerning the presence of VOC contamination in the subsurface. Head space screening is usually applicable to soil samples but can also be modified to include groundwater samples. Although head space screening is not a direct measurement of insitu soil vapors, the procedure is included in this SOP because soil vapors are the actual media being screened or analyzed during headspace screening.

Upon sample retrieval from the subsurface, a small quantity of undisturbed soil (approximately 6 oz.) is removed from the sampler (e.g., hand auger, split-spoon sampler, Shelby tube, etc.) and immediately placed in a sealing (Ziploc®-type) plastic bag. Once sealed in the bag, the sample is gently massaged to break apart any large soil clumps. The bag is then warmed for 15 minutes in order to volatilize the potential contaminants from the soil sample into the head-space of the bag. For consistent readings at a particular site, it is important to warm each sample for the same time period and to approximately the same temperature. For this reason, placing the sample in the passenger compartment of a warm vehicle will provide a consistent ambient temperature for the procedure. Other methods of gently warming the sample can be used as long as the resulting temperature is consistent and the procedure is documented in the field log book. After 15 minutes, the tip of the PID or FID is carefully pushed directly through the plastic bag and a direct reading is obtained of the maximum detection. The PID or FID should be capable of storing the maximum detection value for a given reading. All head-space readings (maximum detection per sample) will be noted on the appropriate soil boring log and/or sample log form.

5.3.2 Pipe Probes

Pipe probes involve the use of a hollow steel tube to collect the soil gas sample. The probes can be passively placed into a predrilled hole or driven to the required depth. The predrilled hole can be made using a slide hammer, bucket auger, electric hammer, or DPT drill rig. The following three soil gas procedures using pipe probes are commonly used for preliminary screening in the field.
The first method described is the simplest and will provide immediate results that may help in the location of subsequent test pits, soil borings, etc. The steps are as described below:

- Drive a 3/4-inch steel pipe into the ground using a slide hammer, electric hammer or equivalent to the desired depth. Generally this will not exceed five feet due to the difficulty in retrieving the pipe.
- Remove the steel pipe from the ground by hand or jack.
- Place a hollow 3/4-inch diameter by 1 foot long steel pipe into the previously created hole and seal off the outside of the pipe with bentonite clay so that no soil gas can escape. Allow 5 minutes for vapors in the hole to reach equilibrium conditions.
- Attach the tip of the PID/FID to the hollow pipe using silicon tubing and collect instrument readings for a minimum of 1 minute, or until the readings peak and begin to decline. Record the highest value in the field notebook along with the time, date, and sample location.

- Decontaminate the steel pipes before moving to the next location to minimize the potential for cross-borehole contamination.

  Note: Any ancillary observations made such as soil type, soil color, depth to water table, etc., should be recorded in the field logbook.

The next method is a variation of the first, but provides a more depth specific sample and uses a Tedlar® bag for the collection of the soil gas.

- Drive an expandable steel drive point (3/4-inches diameter) attached to a 5 foot connecting rod into the ground using a slide or electric hammer or DPT drill rig.
- Retract the rod a sufficient distance to leave a space for the soil gas to enter the drive point (approximately 6 inches). Allow 5 minutes for vapors in the hole to reach equilibrium conditions.
- After the drive point and connecting rod have been retracted, assemble the sampling apparatus by attaching 1/4-inch I.D. polyethylene tubing to the top of the connecting rod.
- Place the tubing to the inlet side of a peristaltic pump and connect the discharge side of the pump to a 1-liter Tedlar® bag.
- Fill the Tedlar® bag twice to adequately purge the sample equipment and ambient air that exists in the bag.
- Collect the soil gas sample in a separate Tedlar® bag as above and then connect it directly to the inlet of a PID/FID.
- Obtain the maximum reading from the PID/FID in ppm and record the data in the field notebook or sample log form.
- Decontaminate the equipment before moving to the next location.

The third procedure is similar to the above, except that the soil gas collected in the Tedlar® bag is analyzed in the field using either a portable or onsite lab gas chromatograph (GC). This procedure can identify specific contaminant(s) of concern. These instruments, though more expensive, can be very sensitive and selective to the contaminant(s) of concern.

5.3.3 Passive Sorbent Samplers

The most common type of sorbent sampler used is known as a Petrex® tube, which consists of activated charcoal chemically fused to the tip of a Curie-point ferromagnetic wire and inserted into a glass tube. The collector is then buried at a depth of 2 to 4 feet in an inverted position with the glass tube acting as a flux chamber for an optimal period of time as determined by the manufacturers recommendations. Sample analysis is by thermal desorption onto a mass spectrometer (MS) or GC.

This method is recommended when the contaminants are unknown and concentrations are expected to be low. Specialized sorbent samplers may require the use of a soil gas subcontractor.

5.3.4 Well Points

Well points can be installed to obtain data on subsurface gas concentrations at depths or areas inaccessible by other monitoring techniques. Single or multiple probes may be installed in a single borehole. Well points are recommended for projects if more than one soil gas sampling event is to occur to monitor contaminant migration versus time. The construction of the well points may vary and may require the use of a conventional drill rig, DPT drill rig, power auger, or hand auger. The need for this type of soil gas survey shall be determined by the Project Manager and site specific conditions. The installation of a soil gas well point is described below:

- Drill the borehole to the desired depth.
- Set the screened well point at the desired depth and backfill with a clean silica sand to approximately 1 foot above the screened length.
- Place soil cuttings or bentonite pellets on top of the sand pack.
- Place 1 to 2 feet of a cement/bentonite grout on top of the bentonite plug.
- If necessary, install a steel protective casing over the well point to prevent damage.
- Place sample outlets inside the protective casing for sampling.

6.0 REFERENCES

New Jersey Department of Environmental Protection and Energy, Field Sampling Procedures Manual, May, 1992.

7.0 RECORDS

A record of all field procedures, tests, and observations must be recorded in the field logbook. Entries should include all pertinent data regarding the soil gas survey. The use of sketches, photographs, and field landmarks will help to supplement the investigation and evaluation.

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		Effective Date 09/03	Revision 3
		Applicability Tetra Tech NUS, Inc.	
		Prepared Earth Sciences Department	
Subject DIRECT PUSH TECHNOLOGY (GEOPROBE®/HYDROPUNCH™)		Approved D. Senovich	

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ATTACHMENTS

1.0 PURPOSE

The purpose of this procedure is to provide general reference information on Direct Push Technology (DPT). DPT is designed to collect soil, groundwater, and soil gas samples without using conventional drilling techniques. The advantage of using DPT over conventional drilling includes the generation of little or no drill cuttings, sampling in locations with difficult accessibility, reduced overhead clearance requirements, no fluid introduction during probing, and typical lower costs per sample than with conventional techniques. Disadvantages include a maximum penetration depth of approximately 15 to 40 feet in dense soils (although it may be as much as 60 to 80 feet in certain types of geological environments), reduced capability of obtaining accurate water-level measurements, and the inability to install permanent groundwater monitoring wells. The methods and equipment described herein are for collection of surface and subsurface soil samples and groundwater samples. Soil gas sampling is discussed in SOP SA-2.4.

2.0 SCOPE

This procedure provides information on proper sampling equipment and techniques for DPT. Review of the information contained herein will facilitate planning of the field sampling effort by describing standard sampling techniques. The techniques described shall be followed whenever applicable, noting that site-specific conditions or project-specific plans may require adjustments in methodology.

3.0 GLOSSARY

Direct Push Technology (DPT) - DPT refers to sampling tools and sensors that are driven directly into the ground without the use of conventional drilling equipment. DPT typically utilizes hydraulic pressure and/or percussion hammers to advance the sampling tools. A primary advantage of DPT over conventional drilling techniques is that DPT results in the generation of little or no investigation derived waste.

Geoprobe® - Geoprobe® is a manufacturer of a hydraulically-powered, percussion/probing machines utilizing DPT to collect subsurface environmental samples. Geoprobe® relies on a relatively small amount of static weight (vehicle) combined with percussion as the energy for advancement of a tool string. The Geoprobe® equipment can be mounted in a multitude of vehicles for access to all types of environmental sites.

HydroPunch™ - HydroPunch™ is a manufacturer of stainless steel and Teflon® sampling tools that are capable of collecting representative groundwater and/or soil samples without requiring the installation of a groundwater monitoring well or conventional soil boring. HydroPunch™ is an example of DPT sampling equipment.

Flame Ionization Detector (FID) - A portable instrument for the measurement of many combustible organic compounds and a few inorganic compounds in air at parts-per million levels. The basis for the detection is the ionization of gaseous species utilizing a flame as the energizing source.

Photo Ionization Detector (PID) - A portable instrument for the measurement of many combustible organic compounds and a few inorganic compounds in air at parts-per million levels. The basis for the detection is the ionization of gaseous species utilizing ultraviolet radiation as the energizing source.

4.0 RESPONSIBILITIES

Project Manager - The Project Manager is responsible for selecting and/or reviewing the appropriate DPT drilling procedure required to support the project objectives.

Field Operations Leader (FOL)- The FOL is primarily responsible for performing the DPT in accordance with the project-specific plan.

5.0 SOIL SAMPLING PROCEDURES

5.1 General

The common methodology for the investigation of the vadose zone is soil boring drilling and soil sampling. However, drilling soil borings can be very expensive. Generally the advantage of DPT for subsurface soil sampling is the reduced cost of disposal of drilling cuttings and shorter sampling times.

5.2 Sampling Equipment

Equipment needed for conducting DPT drilling for subsurface soil sampling includes, but is not limited to, the following:

- Geoprobe® Sampling Kit
- Cut-resistant gloves
- 4-foot x 1.5-inch diameter macrocore sampler
- Probe sampling adapters
- Roto-hammer with 1.5-inch bit
- Disposable acetate liners for soil macrocore sampler
- Cast aluminum or steel drive points
- Geoprobe® AT-660 Series Large Bore Soil Sampler, or equivalent
- Standard decontamination equipment and solutions

For health and safety equipment and procedures, follow the direction provided in the Safe Work Permit in Attachment 1, or the more detailed directions provided in the project's Health and Safety Plan.

5.3 DPT Sampling Methodology

There are several methods for the collection of soil samples using DPT drilling. The most common method is discussed in the following section. Variations of the following method may be conducted upon approval of the Project Manager in accordance with the project-specific plan.

- Macrocore samplers fitted with detachable aluminum or steel drive points are driven into the ground using hydraulic pressure. If there is concrete or pavement over a sampling location, a Roto-hammer is used to drill a minimum 1.5-inch diameter hole through the surface material. A Roto-hammer may also be used if very dense soils are encountered.

- The sampler is advanced continuously in 4-foot intervals or less if desired. No soil cuttings are generated because the soil which is not collected in the sampler is displaced within the formation.

- The sampler is retracted from the hole, and the 4-foot continuous sample is removed from the outer coring tube. The sample is contained within an inner acetate liner.

- Attach the metal trough from the Geoprobe® Sampling Kit firmly to the tail gate of a vehicle. If a vehicle with a tail gate is not available, secure the trough on another suitable surface.

- Place the acetate liner containing the soils in the trough.

- While wearing cut-resistant gloves (constructed of leather or other suitable material), cut the acetate liner through its entire length using the double-bladed knife that accompanies the Geoprobe® Sampling Kit. Then remove the strip of acetate from the trough to gain access to the collected soils. Do not attempt to cut the acetate liner while holding it in your hand.
- Field screen the sample with an FID or PID, and observe/examine the sample (according to SOP GH-1.3). If appropriate, transfer the sample to sample bottles for laboratory analysis. If additional volume is required, push an additional boring adjacent to the first and composite/mix the same interval. Field compositing is usually not acceptable for sample requiring volatile organics analysis.
- Once sampling has been completed, the hole is backfilled with bentonite chips or bentonite cement grout, depending upon project requirements. Asphalt or concrete patch is used to cap holes through paved or concrete areas. All holes should be finished smooth to existing grade.
- In the event the direct push van/truck cannot be driven to a remote location or a sampling location with difficult accessibility, sampling probes may be advanced and sampled manually or with air/electric operated equipment (e.g., jack hammer).
- Sampling equipment is decontaminated prior to collecting the next sample.

6.0 GROUNDWATER SAMPLING PROCEDURES

6.1 General

The most common methodology for the investigation of groundwater is the installation and sampling of permanent monitoring wells. If only groundwater screening is required, the installation and sampling of temporary well points may be performed. The advantage of temporary well point installation using DPT is reduced cost due to no or minimal disposal of drilling cuttings and well construction materials, and shorter installation/times sampling.

Two disadvantages of DPT drilling for well point installation are:

- In aquifers with low yields, well points may have to be sampled without purging or development.
- If volume requirements are high, this method can be time consuming for low yield aquifers.

6.2 Sampling Equipment

Equipment needed for temporary well installation and sampling using DPT includes, but is not limited, to the following:

- 2-foot x 1-inch diameter mill-slotted (0.005 to 0.02-inch) well point
- Connecting rods
- Roto-hammer with 1.5-inch bit
- Mechanical jack
- 1/4-inch OD polyethylene tubing
- 3/8-inch OD polyethylene tubing
- Peristaltic pump
- Standard decontamination equipment and solutions

6.3 DPT Temporary Well Point Installation and Sampling Methodology

There are several methods for the installation and sampling of temporary well points using DPT. The most common methodology is discussed below. Variations of the following method may be conducted upon approval of the Project Manager in accordance with the project specific plan.

- A 2-foot x 1-inch diameter mill-slotted (0.005 to 0.02-inch) well point attached to connecting rods is driven into the ground to the desired depth using a rotary electric hammer or other direct push drill rig. If there is concrete or pavement over a sampling location, a Roto-hammer or electric coring machine is used to drill a hole through the surface material.

- The well point will be allowed to equilibrate for at least 15 minutes, after which a measurement of the static water level will be taken. The initial measurement of the water level will be used to assess the amount of water which is present in the well point and to determine the amount of silt and sand infiltration that may have occurred.

- The well point will be developed using a peristaltic pump and polyethylene tubing to remove silt and sand which may have entered the well point. The well point is developed by inserting polyethylene tubing to the bottom of the well point and lifting and lowering the tubing slightly while the pump is operating. The pump will be operated at a maximum rate of approximately 2 liters per minute. After removal of sediment from the bottom of the well point, the well point will be vigorously pumped at maximum capacity until discharge water is visibly clear and no further sediments are being generated. Measurements of pH, specific conductance, temperature, and turbidity shall be recorded every 5 to 10 minutes during the purging process. After two consistent readings of pH, specific conductance, temperature and turbidity ($\pm$10 percent), the well may be sampled.

- A sample will be collected using the peristaltic pump set at the same or reduced speed as during well development. Samples (with the exception of the samples to be analyzed for volatile organic compounds, VOCs) will be collected directly from the pump discharge. Sample containers for VOCs will be filled by (first shutting off the pump) crimping the discharge end of the sample tubing when filled, removing the inlet end of the sample tubing from the well, suspending the inlet tubing above the vial, and allowing water to fill each vial by gravity flow.

- Once the groundwater sample has been collected, the connecting rods and well point will be removed from the hole with the direct push rig hydraulics. The hole will be backfilled with bentonite chips or bentonite cement grout, depending upon project requirements. Asphalt or concrete patch will be used to cap holes through paved or concrete areas. All holes will be finished smooth to existing grade.

- In the event the direct push van/truck cannot be driven to a remote location or sampling location with difficult accessibility, sampling probes may be advanced and sampled manually or with air/electric-operated equipment (e.g., jack hammer).

- Decontaminate the equipment before moving to the next location.

7.0 RECORDS

A record of all field procedures, tests, and observations must be recorded in the field logbook, boring logs, and sample log sheets, as needed. Entries should include all pertinent data regarding the investigation. The use of sketches and field landmarks will help to supplement the investigation and evaluation.

ATTACHMENT 1
SAFE WORK PERMIT FOR DPT OPERATIONS

Permit No. ____________ Date: ________________ Time: From __________ to ___________

SECTION I: General Job Scope

I. Work limited to the following (description, area, equipment used): **Monitoring well drilling and installation through direct push technology**

II. Required Monitoring Instruments: ____________________

III. Field Crew: ____________________

IV. On-site Inspection conducted ☐ Yes ☐ No Initials of Inspector __________ TtNUS __________

SECTION II: General Safety Requirements (To be filled in by permit issuer)

V. Protective equipment required
Level D ☒ Level B ☐
Level C ☐ Level A ☐
Detailed on Reverse

Respiratory equipment required
Full face APR ☐ Escape Pack ☐
Half face APR ☐ SCBA ☐
SKA-PAC SAR ☐ Bottle Trailer ☐
Skid Rig ☐ None ☒

Level D Minimum Requirements: Sleeved shirt and long pants, safety footwear, and work gloves. Safety glasses, hard hats, and hearing protection will be worn when working near or sampling in the vicinity of the DPT rig.

Modifications/Exceptions.

VI. Chemicals of Concern | Action Level(s) | Response Measures

VII. Additional Safety Equipment/Procedures

Hard-hat	☒ Yes	☐ No	Hearing Protection (Plugs/Muffs)	☒ Yes	☐ No
Safety Glasses	☒ Yes	☐ No	Safety belt/harness	☐ Yes	☒ No
Chemical/splash goggles	☐ Yes	☒ No	Radio	☐ Yes	☒ No
Splash Shield	☐ Yes	☒ No	Barricades	☒ Yes	☐ No
Splash suits/coveralls	☐ Yes	☐ No	Gloves (Type - __________)	☐ Yes	☐ No
Steel toe Work shoes or boots	☒ Yes	☐ No	Work/warming regimen	☐ Yes	☐ No

Modifications/Exceptions: Reflective vests for high traffic areas.

VIII. Procedure review with permit acceptors

	Yes	NA		Yes	NA
Safety shower/eyewash (Location & Use)	☐	☒	Emergency alarms	☐	☐
Daily tail gate meetings	☒	☐	Evacuation routes	☐	☐
Contractor tools/equipment/PPE inspected	☐	☐	Assembly points	☐	☐

IX. Site Preparation

Utility Clearances obtained for areas of subsurface investigation	☐ Yes	☐ No
Physical hazards removed or blockaded	☐ Yes	☐ No
Site control boundaries demarcated/signage	☐ Yes	☐ No

X. Equipment Preparation

	Yes	NA
Equipment drained/depressurized	☐	☒
Equipment purged/cleaned	☐	☒
Isolation checklist completed	☐	☒
Electrical lockout required/field switch tested	☐	☒
Blinds/misalignments/blocks & bleeds in place	☐	☒
Hazardous materials on walls/behind liners considered	☐	☒

XI. Additional Permits required (Hot work, confined space entry). ☐ Yes ☐ No
If yes, complete permit required or contact Health Sciences, Pittsburgh Office

XII. Special instructions, precautions:

Permit Issued by: ____________________ Permit Accepted by: ____________________

TETRA TECH NUS, INC.	**STANDARD OPERATING PROCEDURES**	Number SA-6.1	Page 1 of 11
		Effective Date 02/04	Revision 3
		Applicability Tetra Tech NUS, Inc.	
		Prepared Earth Sciences Department	
Subject NON-RADIOLOGICAL SAMPLE HANDLING		Approved D. Senovich	

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ATTACHMENTS

1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to provide information on sample preservation, packaging, and shipping procedures to be used in handling environmental samples submitted for chemical constituent, biological, or geotechnical analysis. Sample chain-of-custody procedures and other aspects of field documentation are addressed in SOP SA-6.3. Sample identification is addressed in SOP CT-04.

2.0 SCOPE

This procedure describes the appropriate containers to be used for samples depending on the analyses to be performed, and the steps necessary to preserve the samples when shipped off site for chemical analysis.

3.0 GLOSSARY

Hazardous Material - A substance or material which has been determined by the Secretary of Transportation to be capable of posing an unreasonable risk to health, safety, and property when transported in commerce, and which has been so designated. Under 49 CFR, the term includes hazardous substances, hazardous wastes, marine pollutants, and elevated temperature materials, as well as materials designated as hazardous under the provisions of §172.101 and §172.102 and materials that meet the defining criteria for hazard classes and divisions in Part 173. With slight modifications, IATA has adopted DOT "hazardous materials" as IATA "Dangerous Goods."

Hazardous Waste - Any substance listed in 40 CFR, Subpart D (¥261.30 et seq.), or otherwise characterized as ignitable, corrosive, reactive, or toxic (as defined by Toxicity Characteristic Leaching Procedure, TCLP, analysis) as specified under 40 CFR, Subpart C (¥261.20 et seq.), that would be subject to manifest requirements specified in 40 CFR 262. Such substances are defined and regulated by EPA.

Marking - A descriptive name, identification number, instructions, cautions, weight, specification or UN marks, or combination thereof required on outer packaging of hazardous materials.

n.o.i - Not otherwise indicated (may be used interchangeably with n.o.s.).

n.o.s. - Not otherwise specified.

Packaging - A receptacle and any other components or materials necessary for compliance with the minimum packaging requirements of 49 CFR 174, including containers (other than freight containers or overpacks), portable tanks, cargo tanks, tank cars, and multi-unit tank-car tanks to perform a containment function in conformance with the minimum packaging requirements of 49 CFR 173.24(a) & (b).

Placard - Color-coded, pictorial sign which depicts the hazard class symbol and name and which is placed on the side of a vehicle transporting certain hazardous materials.

Common Preservatives:

- Hydrochloric Acid - HCl
- Sulfuric Acid - H_2SO_4
- Nitric Acid - HNO_3
- Sodium Hydroxide - NaOH

Other Preservatives

- Zinc Acetate
- Sodium Thiosulfate - $Na_2S_2O_3$

Normality (N) - Concentration of a solution expressed as equivalent per liter, an equivalent being the amount of a substance containing 1 gram-atom of replaceable hydrogen or its equivalent.

Reportable Quantity (RQ) - For the purposes of this SOP, means the quantity specified in column 3 of the Appendix to DOT 49 CFR §172.101 for any material identified in column 1 of the appendix. A spill greater than the amount specified must be reported to the National Response Center.

Sample - A sample is physical evidence collected from a facility or the environment, which is representative of conditions at the location and time of collection.

4.0 RESPONSIBILITIES

Field Operations Leader - Directly responsible for the bottling, preservation, labeling, packaging, shipping, and custody of samples up to and including release to the shipper.

Field Samplers - Responsible for initiating the Chain-of-Custody Record (per SOP SA-6.3), implementing the packaging and shipping requirements, and maintaining custody of samples until they are relinquished to another custodian or to the shipper.

5.0 PROCEDURES

Sample identification, labeling, documentation, and chain-of-custody are addressed by SOP SA-6.3.

5.1 Sample Containers

Different types of chemicals react differently with sample containers made of various materials. For example, trace metals adsorb more strongly to glass than to plastic, whereas many organic chemicals may dissolve various types of plastic containers. Attachments A and B show proper containers (as well as other information) per 40 CFR 136. In general, the sample container shall allow approximately 5-10 percent air space ("ullage") to allow for expansion/vaporization if the sample warms during transport. However, for collection of volatile organic compounds, head space shall be omitted. The analytical laboratory will generally provide certified-clean containers for samples to be analyzed for chemical constituents. Shelby tubes or other sample containers are generally provided by the driller for samples requiring geotechnical analysis. Sufficient lead time shall be allowed for a delivery of sample container orders. Therefore, it is critical to use the correct container to maintain the integrity of the sample prior to analysis.

Once opened, the container must be used at once for storage of a particular sample. Unused but opened containers are to be considered contaminated and must be discarded. Because of the potential for introduction of contamination, they cannot be reclosed and saved for later use. Likewise, any unused containers which appear contaminated upon receipt, or which are found to have loose caps or a missing Teflon liner (if required for the container), shall be discarded.

5.2 Sample Preservation

Many water and soil samples are unstable and therefore require preservation to prevent changes in either the concentration or the physical condition of the constituent(s) requiring analysis. Although complete and irreversible preservation of samples is not possible, preservation does retard the chemical and biological changes that inevitably take place after the sample is collected. Preservation techniques are usually limited to pH control, chemical addition(s), and refrigeration/ freezing (certain biological samples only).

5.2.1 Overview

The preservation techniques to be used for various analytes are listed in Attachments A and B. Reagents required for sample preservation will either be added to the sample containers by the laboratory prior to their shipment to the field or be added in the field (in a clean environment). Only high purity reagents shall be used for preservation. In general, aqueous samples of low-concentration organics (or soil samples of low- or medium-concentration organics) are cooled to 4°C. Medium-concentration aqueous samples, high-hazard organic samples, and some gas samples are typically not preserved. Low-concentration aqueous samples for metals are acidified with HNO_3, whereas medium-concentration and high-hazard aqueous metal samples are not preserved. Low- or medium-concentration soil samples for metals are cooled to 4°C, whereas high-hazard samples are not cooled.

The following subsections describe the procedures for preparing and adding chemical preservatives. Attachments A and B indicate the specific analytes which require these preservatives.

The FOL is responsible for ensuring that an accurate Chemical Inventory is created and maintained for all hazardous chemicals brought to the work site (see Section 5 of the TtNUS Health and Safety Guidance Manual). Furthermore, the FOL must ensure that a corresponding Material Safety Data Sheet (MSDS) is collected for every substance entered on the site Chemical Inventory, and that all persons using/handling/ disposing of these substances review the appropriate MSDS for substances they will work with. The Chemical Inventory and the MSDSs must be maintained at each work site in a location and manner where they are readily-accessible to all personnel.

5.2.2 Preparation and Addition of Reagents

Addition of the following acids or bases may be specified for sample preservation; these reagents shall be analytical reagent (AR) grade or purer and shall be diluted to the required concentration with deionized water before field sampling commences. To avoid uncontrolled reactions, be sure to Add Acid to water (not vice versa). A dilutions guide is provided below.

Acid/Base	Dilution	Concentration	Estimated Amount Required for Preservation
Hydrochloric Acid (HCl)	1 part concentrated HCl: 1 part double-distilled, deionized water	6N	5-10 mL
Sulfuric Acid (H_2SO_4)	1 part concentrated H_2SO_4: 1 part double-distilled, deionized water	18N	2 - 5 mL
Nitric Acid (HNO_3)	Undiluted concentrated HNO_3	16N	2 - 5 mL
Sodium Hydroxide (NaOH)	400 grams solid NaOH dissolved in 870 mL double-distilled, deionized water; yields 1 liter of solution	10N	2 mL

The amounts required for preservation shown in the above table assumes proper preparation of the preservative and addition of the preservative to one liter of aqueous sample. This assumes that the sample is initially at pH 7, is poorly buffered, and does not contain particulate matter; as these conditions vary, more preservative may be required. Consequently, the final sample pH must be checked using narrow-range pH paper, as described in the generalized procedure detailed below:

- Pour off 5-10 mL of sample into a dedicated, clean container. Use some of this sample to check the initial sample pH using wide range (0-14) pH paper. Never dip the pH paper into the sample; always apply a drop of sample to the pH paper using a clean stirring rod or pipette.

- Add about one-half of the estimated preservative required to the original sample bottle. Cap and invert gently several times to mix. Check pH (as described above) using medium range pH paper (pH 0-6 or pH 7.5-14, as applicable).

- Cap sample bottle and seal securely.

Additional considerations are discussed below:

- To test if ascorbic acid must be used to remove oxidizing agents present in the sample before it can be properly preserved, place a drop of sample on KI-starch paper. A blue color indicates the need for ascorbic acid addition.

  If required, add a few crystals of ascorbic acid to the sample and retest with the KI-starch paper. Repeat until a drop of sample produces no color on the KI-starch paper. Then add an additional 0.6 grams of ascorbic acid per each liter of sample volume.

  Continue with proper base preservation of the sample as described above.

- Samples for sulfide analysis must be treated by the addition of 4 drops (0.2 mL) of 2N zinc acetate solution per 100 ml of sample.

  The 2N zinc acetate solution is made by dissolving 220 grams of zinc acetate in 870 mL of double-distilled, deionized water to make 1 liter of solution.

  The sample pH is then raised to 9 using the NaOH preservative.

- Sodium thiosulfate must be added to remove residual chlorine from a sample. To test the sample for residual chlorine use a field test kit specially made for this purpose.

  If residual chlorine is present, add 0.08 grams of sodium thiosulfate per liter of sample to remove the residual chlorine.

  Continue with proper acidification of the sample as described above.

For biological samples, 10% buffered formalin or isopropanol may also be required for preservation. Questions regarding preservation requirements should be resolved through communication with the laboratory before sampling begins.

5.3 Field Filtration

At times, field-filtration may be required to provide for the analysis of dissolved chemical constituents. Field-filtration must be performed prior to the preservation of samples as described above. General procedures for field filtration are described below:

- The sample shall be filtered through a non-metallic, 0.45-micron membrane filter, immediately after collection. The filtration system shall consist of dedicated filter canister, dedicated tubing, and a peristaltic pump with pressure or vacuum pumping squeeze action (since the sample is filtered by mechanical peristalsis, the sample travels only through the tubing).

- To perform filtration, thread the tubing through the peristaltic pump head. Attach the filter canister to the discharge end of the silicon tubing (note flow direction arrow); attach the aqueous sample container to the intake end of the silicon tubing. Turn the peristaltic pump on and perform filtration. Run approximately 100 ml of sample through the filter and discard prior to sample collection.

- Continue by preserving the filtrate (contained in the filter canister), as applicable and generally described above.

5.4 Sample Packaging and Shipping

Only employees who have successfully completed the TtNUS "Shipping Hazardous Materials" training course are authorized to package and ship hazardous substances. These trained individuals are responsible for performing shipping duties in accordance with this training.

Samples collected for shipment from a site shall be classified as either environmental or hazardous material samples. Samples from drums containing materials other than Investigative Derived Waste (IDW) and samples obtained from waste piles or bulk storage tanks are generally shipped as hazardous materials. A distinction must be made between the two types of samples in order to:

- Determine appropriate procedures for transportation of samples (if there is any doubt, a sample shall be considered hazardous and shipped accordingly.)

- Protect the health and safety of transport and laboratory personnel receiving the samples (special precautions are used by the shipper and at laboratories when hazardous materials are received.)

Detailed procedures for packaging environmental samples are outlined in the remainder of this section.

5.4.1 Environmental Samples

Environmental samples are packaged as follows:

- Place properly identified sample container, with lid securely fastened, in a plastic bag (e.g. Ziploc baggie), and seal the bag.

- Place sample in a cooler constructed of sturdy material which has been lined with a large, plastic bag (e.g. "garbage" bag). Drain plugs on coolers must be taped shut.

- Pack with enough cushioning materials such as bubble wrap (shoulders of bottles must be iced if required) to minimize the possibility of the container breaking.

- If cooling is required (see Attachments A and B), place ice around sample container shoulders, and on top of packing material (minimum of 8 pounds of ice for a medium-size cooler).

- Seal (i.e., tape or tie top in knot) large liner bag.

- The original (top, signed copy) of the COC form shall be placed inside a large Ziploc-type bag and taped inside the lid of the shipping cooler. If multiple coolers are sent but are included on one COC form, the COC form should be sent with the cooler containing the vials for VOC analysis. The COC form should then state how many coolers are included with that shipment.

- Close and seal outside of cooler as described in SOP SA-6.3. Signed custody seals must be used.

Coolers must be marked as containing "Environmental Samples." The appropriate side of the container must be marked "This End Up" and arrows placed appropriately. No DOT marking or labeling is required; there are no DOT restrictions on mode of transportation.

6.0 REFERENCES

American Public Health Association, 1981. Standard Methods for the Examination of Water and Wastewater, 15th Edition. APHA, Washington, D.C.

International Air Transport Association (latest issue). Dangerous Goods Regulations, Montreal, Quebec, Canada.

U.S. Department of Transportation (latest issue). Hazardous Materials Regulations, 49 CFR 171-177.

U.S. EPA, 1984. "Guidelines Establishing Test Procedures for the Analysis of Pollutants under Clean Water Act." Federal Register, Volume 49 (209), October 26, 1984, p. 43234.

U.S. EPA, 1979. Methods for Chemical Analysis of Water and Wastes. EPA-600/4-79-020, U.S. EPA-EMSL, Cincinnati, Ohio.

ATTACHMENT A

GENERAL SAMPLE CONTAINER AND PRESERVATION REQUIREMENTS

Sample Type and Concentration		Container[1]	Sample Size	Preservation[2]	Holding Time[2]
WATER					
Organics (GC&GC/MS)	VOC Low	Borosilicate glass	2 x 40 mL	Cool to 4°C HCl to ≤ 2	14 days[9]
	Extractables (Low SVOCs and pesticide/PCBs)	Amber glass	2x2 L or 4x1 L	Cool to 4°C	7 days to extraction; 40 days after extraction
	Extractables (Medium SVOCs and pesticide/PCBs)	Amber glass	2x2 L or 4x1 L	None	7 days to extraction; 40 days after extraction
Inorganics	Metals Low	High-density polyethylene	1 L	HNO_3 to pH ≤2	6 months (Hg-28 days)
	Medium	Wide-mouth glass	16 oz.	None	6 months
	Cyanide Low	High-density polyethylene	1 L	NaOH to pH>12	14 days
	Cyanide Medium	Wide-mouth glass	16 oz.	None	14 days
Organic/ Inorganic	High Hazard	Wide-mouth glass	8 oz.	None	14 days
SOIL					
Organics (GC&GC/MS)	VOC	EnCore Sampler	(3) 5 g Samplers	Cool to 4°C	48 hours to lab preservation
	Extractables (Low SVOCs and pesticides/PCBs)	Wide-mouth glass	8 oz.	Cool to 4°C	14 days to extraction; 40 days after extraction
	Extractables (Medium SVOCs and pesticides/PCBs)	Wide-mouth glass	8 oz.	Cool to 4°C	14 days to extraction; 40 days after extraction
Inorganics	Low/Medium	Wide-mouth glass	8 oz.	Cool to 4°C	6 months (Hg - 28 days) Cyanide (14 days)
Organic/Inorganic	High Hazard	Wide-mouth glass	8 oz.	None	NA
Dioxin/Furan	All	Wide-mouth glass	4 oz.	None	35 days until extraction; 40 days after extraction
TCLP	All	Wide-mouth glass	8 oz.	None	7 days until preparation; analysis as per fraction
AIR					
Volatile Organics	Low/Medium	Charcoal tube -- 7 cm long, 6 mm OD, 4 mm ID	100 L air	Cool to 4°C	5 days recommended

1 All glass containers should have Teflon cap liners or septa.
2 See Attachment E. Preservation and maximum holding time allowances per 40 CFR 136.

ATTACHMENT B

ADDITIONAL REQUIRED CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES

Parameter Number/Name	Container(1)	Preservation(2)(3)	Maximum Holding Time(4)
INORGANIC TESTS:			
Acidity	P, G	Cool, 4°C	14 days
Alkalinity	P, G	Cool, 4°C	14 days
Ammonia - Nitrogen	P, G	Cool, 4°C; H_2SO_4 to pH 2	28 days
Biochemical Oxygen Demand (BOD)	P, G	Cool, 4°C	48 hours
Bromide	P, G	None required	28 days
Chemical Oxygen Demand (COD)	P, G	Cool, 4°C; H_2SO_4 to pH 2	28 days
Chloride	P, G	None required	28 days
Chlorine, Total Residual	P, G	None required	Analyze immediately
Color	P, G	Cool, 4°C	48 hours
Cyanide, Total and Amenable to Chlorination	P, G	Cool, 4°C; NaOH to pH 12; 0.6 g ascorbic acid(5)	14 days(6)
Fluoride	P	None required	28 days
Hardness	P, G	HNO_3 to pH 2; H_2SO_4 to pH 2	6 months
Total Kjeldahl and Organic Nitrogen	P, G	Cool, 4°C; H_2SO_4 to pH 2	28 days
Nitrate - Nitrogen	P, G	None required	48 hours
Nitrate-Nitrite - Nitrogen	P, G	Cool, 4°C; H_2SO_4 to pH 2	28 days
Nitrite - Nitrogen	P, G	Cool, 4°C	48 hours
Oil & Grease	G	Cool, 4°C; H_2SO_4 to pH 2	28 days
Total Organic Carbon (TOC)	P, G	Cool, 4°C; HCl or H_2SO_4 to pH 2	28 days
Orthophosphate	P, G	Filter immediately; Cool, 4°C	48 hours
Oxygen, Dissolved-Probe	G Bottle & top	None required	Analyze immediately
Oxygen, Dissolved-Winkler	G Bottle & top	Fix on site and store in dark	8 hours
Phenols	G	Cool, 4°C; H_2SO_4 to pH 2	28 days
Phosphorus, Total	P, G	Cool, 4°C; H_2SO_4 to pH 2	28 days
Residue, Total	P, G	Cool, 4°C	7 days
Residue, Filterable (TDS)	P, G	Cool, 4°C	7 days
Residue, Nonfilterable (TSS)	P, G	Cool, 4°C	7 days
Residue, Settleable	P, G	Cool, 4°C	48 hours
Residue, Volatile (Ash Content)	P, G	Cool, 4°C	7 days
Silica	P	Cool, 4°C	28 days
Specific Conductance	P, G	Cool, 4°C	28 days
Sulfate	P, G	Cool, 4°C	28 days

ATTACHMENT B
ADDITIONAL REQUIRED CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES
PAGE TWO

Parameter Number/Name	Container[1]	Preservation[2][3]	Maximum Holding Time[4]
INORGANIC TESTS (Cont'd):			
Sulfide	P, G	Cool, 4°C; add zinc acetate plus sodium hydroxide to pH 9	7 days
Sulfite	P, G	None required	Analyze immediately
Turbidity	P, G	Cool, 4°C	48 hours
METALS:[7]			
Chromium VI (Hexachrome)	P, G	Cool, 4°C	24 hours
Mercury (Hg)	P, G	HNO_3 to pH 2	28 days
Metals, except Chromium VI and Mercury	P, G	HNO_3 to pH 2	6 months
ORGANIC TESTS:[8]			
Purgeable Halocarbons	G, Teflon-lined septum	Cool, 4°C; 0.008% $Na_2S_2O_3$[5]	14 days
Purgeable Aromatic Hydrocarbons	G, Teflon-lined septum	Cool, 4°C; 0.008% $Na_2S_2O_3$[5] HCl to pH 2 [9]	14 days
Acrolein and Acrylonitrile	G, Teflon-lined septum	Cool, 4°C; 0.008% $Na_2S_2O_3$[5] adjust pH to 4-5 [10]	14 days
Phenols[11]	G, Teflon-lined cap	Cool, 4°C; 0.008% $Na_2S_2O_3$[5]	7 days until extraction; 40 days after extraction
Benzidines[11], [12]	G, Teflon-lined cap	Cool, 4°C; 0.008% $Na_2S_2O_3$[5]	7 days until extraction[13]
Phthalate esters[11]	G, Teflon-lined cap	Cool, 4°C	7 days until extraction; 40 days after extraction
Nitrosamines[11], [14]	G, Teflon-lined cap	Cool, 4°C; store in dark; 0.008% $Na_2S_2O_3$[5]	7 days until extraction; 40 days after extraction
PCBs[11]	G, Teflon-lined cap	Cool, 4°C	7 days until extraction; 40 days after extraction
Nitroaromatics & Isophorone[11]	G, Teflon-lined cap	Cool, 4°C; 0.008% $Na_2S_2O_3$[5]; store in dark	7 days until extraction; 40 days after extraction
Polynuclear Aromatic Hydrocarbons (PAHs)[11],[14]	G, Teflon-lined cap	Cool, 4°C; 0.008% $Na_2S_2O_3$[5]; store in dark	7 days until extraction; 40 days after extraction
Haloethers[11]	G, Teflon-lined cap	Cool, 4°C; 0.008% $Na_2S_2O_3$[5]	7 days until extraction; 40 days after extraction
Dioxin/Furan (TCDD/TCDF)[11]	G, Teflon-lined cap	Cool, 4°C; 0.008% $Na_2S_2O_3$[5]	7 days until extraction; 40 days after extraction

ATTACHMENT B
ADDITIONAL REQUIRED CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES
PAGE THREE

(1) Polyethylene (P): generally 500 ml or Glass (G): generally 1L.

(2) Sample preservation should be performed immediately upon sample collection. For composite chemical samples each aliquot should be preserved at the time of collection. When use of an automated sampler makes it impossible to preserve each aliquot, then chemical samples may be preserved by maintaining at 4°C until compositing and sample splitting is completed.

(3) When any sample is to be shipped by common carrier or sent through the United States Mail, it must comply with the Department of Transportation Hazardous Materials Regulations (49 CFR Part 172).

(4) Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still be considered valid. Samples may be held for longer periods only if the permittee, or monitoring laboratory, has data on file to show that the specific types of samples under study are stable for the longer periods, and has received a variance from the Regional Administrator.

(5) Should only be used in the presence of residual chlorine.

(6) Maximum holding time is 24 hours when sulfide is present. Optionally, all samples may be tested with lead acetate paper before pH adjustments are made to determine if sulfide is present. If sulfide is present, it can be removed by the addition of cadmium nitrate powder until a negative spot test is obtained. The sample is filtered and then NaOH is added to pH 12.

(7) Samples should be filtered immediately on site before adding preservative for dissolved metals.

(8) Guidance applies to samples to be analyzed by GC, LC, or GC/MS for specific compounds.

(9) Sample receiving no pH adjustment must be analyzed within 7 days of sampling.

(10) The pH adjustment is not required if acrolein will not be measured. Samples for acrolein receiving no pH adjustment must be analyzed within 3 days of sampling.

(11) When the extractable analytes of concern fall within a single chemical category, the specified preservative and maximum holding times should be observed for optimum safeguard of sample integrity. When the analytes of concern fall within two or more chemical categories, the sample may be preserved by cooling to 4°C, reducing residual chlorine with 0.008% sodium thiosulfate, storing in the dark, and adjusting the pH to 6-9; samples preserved in this manner may be held for 7 days before extraction and for 40 days after extraction. Exceptions to this optional preservation and holding time procedure are noted in footnote 5 (re: the requirement for thiosulfate reduction of residual chlorine) and footnotes 12, 13 (re: the analysis of benzidine).

(12) If 1,2-diphenylthydrazine is likely to be present, adjust the pH of the sample to 4.0±0.2 to prevent rearrangement to benzidine.

(13) Extracts may be stored up to 7 days before analysis if storage is conducted under an inert (oxidant-free) atmosphere.

(14) For the analysis of diphenylnitrosamine, add 0.008% $Na_2S_2O_3$ and adjust pH to 7-10 with NaOH within 24 hours of sampling.

(15) The pH adjustment may be performed upon receipt at the laboratory and may be omitted if the samples are extracted within 72 hours of collection. For the analysis of aldrin, add 0.008% $Na_2S_2O_3$.

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		Effective Date: 03/09/09	Revision: 3
		Applicability: Tetra Tech NUS, Inc.	
		Prepared: Earth Sciences Department	
Subject: FIELD DOCUMENTATION		Approved: Tom Johnston	

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1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to identify and designate the field data record forms, logs, and reports generally initiated and maintained for documenting Tetra Tech NUS, Inc. (TtNUS) field activities.

2.0 SCOPE

Documents presented within this SOP (or equivalents) shall be used for all TtNUS field activities, as applicable. Other or additional documents may be required by specific client contracts or project planning documents.

3.0 GLOSSARY

None.

4.0 RESPONSIBILITIES AND PERSONNEL QUALIFICATIONS

Project Manager (PM) - The PM is responsible for obtaining hardbound controlled-distribution logbooks (from the appropriate source), as needed. In addition, the Project Manager is responsible for placing all field documentation used in site activities (i.e., records, field reports, sample data sheets, field notebooks, and the site logbook) in the project's central file upon the completion of field work.

Field Operations Leader (FOL) - The FOL is responsible for ensuring that the site logbook, notebooks, and all appropriate and current forms and field reports included in this SOP (and any additional forms required by the contract) are correctly used, accurately filled out, and completed in the required time frame.

General personnel qualifications for field documentation activities include the following:

- Occupational Safety and Health Administration (OSHA) 40-hour and applicable refresher training.
- Capability of performing field work under the expected physical and environmental (i.e., weather) conditions.
- Familiarity with appropriate procedures for documentation, handling, packaging, and shipping.

5.0 PROCEDURES

5.1 SITE LOGBOOK

5.1.1 General

The site logbook is a hard-bound, paginated, controlled-distribution record book in which all major on-site activities are documented. At a minimum, record or reference the following activities/events (daily) in the site logbook:

- All field personnel present
- Arrival/departure times and names of site visitors
- Times and dates of health and safety training
- Arrival/departure times of equipment
- Times and dates of equipment calibration

- Start and/or completion of borehole, trench, monitoring well installation activities, etc.
- Daily on-site activities
- Sample pickup information
- Health and safety issues (level of protection, personal protective equipment [PPE], etc.)
- Weather conditions

Maintain a site logbook for each project and initiate it at the start of the first on-site activity (e.g., site visit or initial reconnaissance survey). Make entries every day that on-site activities take place involving TtNUS or subcontractor personnel. Upon completion of the fieldwork, provide the site logbook to the PM or designee for inclusion in the project's central file.

Record the following information on the cover of each site logbook:

- Project name
- TtNUS project number
- Sequential book number
- Start date
- End date

Information recorded daily in the site logbook need not be duplicated in other field notebooks (see Section 5.2) but must summarize the contents of these other notebooks and refer to specific page locations in these notebooks for detailed information (where applicable). An example of a typical site logbook entry is shown in Attachment A.

If measurements are made at any location, either record the measurements and equipment used in the site logbook or reference the field notebook in which the measurements are recorded (see Attachment A).

Make all logbook, notebook, and log sheet entries in indelible ink (black pen is preferred). No erasures are permitted. If an incorrect entry is made, cross out the entry with a single strike mark, initial, and date it. At the completion of entries by any individual, the logbook pages used must be signed and dated by the person making the entries. The site logbook must also be signed by the FOL at the end of each day.

5.1.2 Photographs

Sequentially number movies, slides, or photographs taken of a site or any monitoring location to correspond to logbook/notebook entries. Enter the name of the photographer, date, time, site location, site description, and weather conditions in the logbook/notebook as the photographs are taken. A series entry may be used for rapid-sequence photographs. The photographer is not required to record the aperture settings and shutter speeds for photographs taken within the normal automatic exposure range. However, special lenses, films, filters, and other image-enhancement techniques must be noted in the logbook/notebook. If possible, such techniques shall be avoided because they can adversely affect the accuracy of photographs. Chain-of-custody procedures depend on the subject matter, type of camera (digital or film), and the processing it requires. Follow chain-of-custody procedures for film used for aerial photography, confidential information, or criminal investigation. After processed, consecutively number the slides of photographic prints and label them according to the logbook/notebook descriptions. Docket the site photographs and associated negatives and/or digitally saved images to compact disks into the project's central file.

5.2 <u>FIELD NOTEBOOKS</u>

Key field team personnel may maintain a separate dedicated field notebook to document the pertinent field activities conducted directly under their supervision. For example, on large projects with multiple investigative sites and varying operating conditions, the Health and Safety Officer may elect to maintain a separate field notebook. Where several drill rigs are in operation simultaneously, each site geologist assigned to oversee a rig must maintain a field notebook.

5.3 FIELD FORMS

All TtNUS field forms (see list in Section 6.0 of this SOP) can be found on the company's intranet site (http://intranet.ttnus.com) under Field Log Sheets. Forms may be altered or revised for project-specific needs, subject to client approval. Care must be taken to ensure that all essential information can be documented. Guidelines for completing these forms can be found in the related sampling SOPs.

5.3.1 Sample Collection, Labeling, Shipment, Request for Analysis, and Field Test Results

5.3.1.1 Sample Log Sheet

Sample log sheets are used to record specified types of data while sampling. The data recorded on these sheets are useful in describing the sample as well as pointing out any problems, difficulties, or irregularities encountered during sampling. Complete a sample log sheet for each sample obtained, including field quality control (QC) samples.

5.3.1.2 Sample Label

A typical sample label is illustrated in Attachment B. Complete the required information on the adhesive labels and apply them to every sample container. Obtain sample labels from the appropriate program/project source, request that they be electronically generated in house, or request them the laboratory subcontractor.

5.3.1.3 Chain-of-Custody Record

The chain-of-custody record is a multi-part form that is initiated as samples are acquired and accompanies a sample (or group of samples) as they are transferred from person to person. This form must be used as follows for any samples collected for chemical or geotechnical analysis whether the analyses are performed on site or off site:

- Retain one carbonless copy of the completed chain-of custody form in the field.
- Send one copy is sent to the PM (or designee)
- Send the original to the laboratory with the associated samples. Place the original (top, signed copy) of the chain-of custody form inside a large Ziploc®-type bag taped inside the lid of the shipping cooler. If multiple coolers are sent but are included on one chain-of custody form, send the form with the cooler containing vials for volatile organic compound (VOC) analysis or the cooler with the air bill attached. Indicate on the air bill how many coolers are included with that shipment.

An example of a chain-of-custody form is provided as Attachment C. After the samples are received at the laboratory, the sample cooler and contents are checked and any problems are noted on the enclosed chain-of custody form (any discrepancies between the sample labels and chain-of custody form and any other problems that are noted are resolved through communication between the laboratory point-of-contact and the TtNUS PM). The chain-of custody form is signed and copied. The laboratory will retain the copy, and the original becomes part of the samples' corresponding analytical data package.

5.3.1.4 Chain-of-Custody Seal

Attachment D is an example of a custody seal. The custody seal is an adhesive-backed label that is part of a chain-of-custody process and is used to prevent tampering with samples after they have been collected in the field and sealed in coolers for transport to the laboratory. Sign and date custody seals and affix them across the lid and body of each cooler (front and back) containing environmental samples (see SOP SA-6.1). Obtain custody seals from the laboratory (if available) or purchase them from a supplier.

5.3.1.5 Geochemical Parameters Log Sheets

Complete Field Analytical Log Sheets to record geochemical and/or natural attenuation field test results.

5.3.2 Hydrogeological and Geotechnical Forms

5.3.2.1 Groundwater Level Measurement Sheet

Complete a Groundwater Level Measurement Sheet for each round of water level measurements made at a site.

5.3.2.2 Data Sheet for Pumping Test

During the performance of a pumping test (or an in-situ hydraulic conductivity test), a large amount of data must be recorded, often within a short time period. Use a Pumping Test Data Sheet to facilitate this task by standardizing the data collection format for the pumping well and observation wells, and allowing the time interval for collection to be established in advance.

5.3.2.3 Packer Test Report Form

Complete a Packer Test Report Form for each well at which a packer test is conducted.

5.3.2.4 Boring Log

Complete a Summary Log of Boring, or Boring Log for each soil boring performed to document the materials encountered, operation and driving of casing, and locations/depths of samples collected. In addition, if volatile organics are monitored on cores, samples, cuttings from the borehole, or breathing zone, (using a photoionization detector [PID] or flame ionization detector [FID]), enter these readings on the boring log at the appropriate depth. When they become available, enter the laboratory sample number, concentrations of key contaminants, or other pertinent information in the "Remarks" column. This feature allows direct comparison of contaminant concentrations with soil characteristics.

5.3.2.5 Monitoring Well Construction Details Form

Complete a Monitoring Well Construction Details Form for every monitoring well, piezometer, or temporary well point installed. This form contains specific information on length and type of well riser pipe and screen, backfill, filter pack, annular seal and grout characteristics, and surface seal characteristics. This information is important in evaluating the performance of the monitoring well, particularly in areas where water levels show temporal variation or where there are multiple (immiscible) phases of contaminants. Depending on the type of monitoring well (in overburden or bedrock, stick-up or flush mount), different forms are used.

5.3.2.6 Test Pit Log

When a test pit or trench is constructed for investigative or sampling purposes, a Test Pit Log must be filled out by the responsible field geologist or sampling technician.

5.3.2.7 Miscellaneous Monitoring Well Forms

Miscellaneous monitoring well forms that may be required on a project-specific basis include the Monitoring Well Materials Certificate of Conformance and Monitoring Well Development Record. Use a Monitoring Well Materials Certificate of Conformance to document all materials utilized during each monitoring well installation. Use a Monitoring Well Development Record to document all well development activities.

5.3.2.8 Miscellaneous Field Forms – Quality Assurance and Checklists

Miscellaneous field forms/checklists forms that may be required on a project-specific basis include the following:

- Container Sample and Inspection Sheet – use this form when a container (drum, tank, etc.) is sampled and/or inspected.

- QA Sample Log Sheet – use this form when a QA sample such as an equipment rinsate blank, source blank, etc. is collected.

- Field Task Modification Request (FTMR) – use this form to document deviations from the project planning documents. The FOL is responsible for initiating the FTMRs. Maintain copies of all FTMRs with the on-site planning documents, and place originals in the final evidence file.

- Field Project Daily Activities Checklist and Field Project Pre-Mobilization Checklist – used these during both the planning and field effort to ensure that all necessary tasks are planned for and completed. These two forms are not requirements but are useful tools for most field work.

5.3.3 Equipment Calibration and Maintenance Form

The calibration or standardization of monitoring, measuring, or test equipment is necessary to ensure the proper operation and response of the equipment, to document the accuracy, precision, or sensitivity of the measurements, and determine if correction should be applied to the readings. Some items of equipment require frequent calibration, others infrequent. Some are calibrated by the manufacturer, others by the user.

Each instrument requiring calibration has its own Equipment Calibration Log, which documents that the manufacturer's instructions were followed for calibration of the equipment, including frequency and type of standard or calibration device. Maintain an Equipment Calibration Log for each electronic measuring device used in the field; make entries for each day the equipment is used or in accordance with manufacturer recommendations.

5.4 FIELD REPORTS

The primary means of recording on-site activities is the site logbook. Other field notebooks may also be maintained. These logbooks and notebooks (and supporting forms) contain detailed information required for data interpretation or documentation but are not easily used for tracking and reporting of progress. Furthermore, the field logbook/notebooks remain on site for extended periods of time and are thus not accessible for timely review by project management. Other reports useful for tracking and reporting the progress of field activities are described below.

5.4.1 Daily Activities Report

To provide timely oversight of on-site contractors, complete and submit Daily Activities Reports (DARs) as described below.

5.4.1.1 Description

The DAR documents the activities and progress for each day's field work. Complete this report on a daily basis whenever there are drilling, test pitting, well construction, or other related activities occurring that involve subcontractor personnel. These sheets summarize the work performed and form the basis of payment to subcontractors. The DAR form can be found on the TtNUS intranet site.

5.4.1.2 Responsibilities

It is the responsibility of the rig geologist to complete the DAR and obtain the driller's signature acknowledging that the times and quantities of material entered are correct.

5.4.1.3 Submittal and Approval

At the end of the shift, the rig geologist must submit the DAR to the FOL for review and filing. The Daily Activities Report is not a formal report and thus requires no further approval. The DARs are retained by the FOL for use in preparing the site logbook and in preparing weekly status reports for submission to the PM.

5.4.2 Weekly Status Reports

To facilitate timely review by project management, photocopies of logbook/notebook entries may be made for internal use.

In addition to those described herein, other summary reports may also be contractually required.

All TtNUS field forms can be found on the company's intranet site at http://intranet.ttnus.com under Field Log Sheets.

6.0 LISTING OF FIELD FORMS ON THE TtNUS INTRANET SITE

- Boring Log
- Container Sample and Inspection Sheet
- Daily Activities Checklist
- Daily Activities Record
- Equipment Calibration Log
- Field Task Modification Request
- Field Analytical Log sheet - Geochemical Parameters
- Groundwater Level Measurement Sheet
- Groundwater Sample Log Sheet
- Hydraulic Conductivity Test Data Sheet
- Low Flow Purge Data Sheet
- Bedrock Monitoring Well Construction (Stick Up)
- Bedrock Monitoring Well Construction Flush Mount
- Bedrock Monitoring Well Construction Open Hole
- Confining Layer Monitoring Well Construction
- Monitoring Well Development Record

- Monitoring Well Materials Certificate of Conformance
- Overburden Monitoring Well Construction Flush Mount
- Overburden Monitoring Well Construction Stick Up
- Packer Test Report Form
- Pumping Test Data Sheet
- QA Sample Log Sheet
- Soil/Sediment Sample Log Sheet
- Surface Water Sample Log Sheet
- Test Pit Log
- Field Project Pre-Mobilization Checklist

ATTACHMENT A
TYPICAL SITE LOGBOOK ENTRY

START TIME: ______________________________ DATE: ______________________________

SITE LEADER: ____
PERSONNEL:

TtNUS	DRILLER	SITE VISITORS
____________	____________	____________
____________	____________	____________
____________	____________	____________

WEATHER: Clear, 68°F, 2-5 mph wind from SE

ACTIVITIES:

1. Steam jenney and fire hoses were set up.
2. Drilling activities at well ____ resumes. Rig geologist was ______________. See Geologist's Notebook, No. 1, page 29-30, for details of drilling activity. Sample No. 123-21-S4 collected; see sample logbook, page 42. Drilling activities completed at 11:50 and a 4-inch stainless steel well installed. See Geologist's Notebook, No. 1, page 31, and well construction details for well ______.
3. Drilling rig No. 2 steam-cleaned at decontamination pit. Then set up at location of well _______.
4. Well ______ drilled. Rig geologist was ____________________. See Geologist's Notebook, No. 2, page ____ for details of drilling activities. Sample numbers 123-22-S1, 123-22-S2, and 123-22-S3 collected; see sample logbook, pages 43, 44, and 45.
5. Well _____ was developed. Seven 55-gallon drums were filled in the flushing stage. The well was then pumped using the pitcher pump for 1 hour. At the end of the hour, water pumped from well was "sand free."
6. EPA remedial project manger arrives on site at 14:25 hours.
7. Large dump truck arrives at 14:45 and is steam-cleaned. Backhoe and dump truck set up over test pit _________.
8. Test pit _______ dug with cuttings placed in dump truck. Rig geologist was _______________. See Geologist's Notebook, No. 1, page 32, for details of test pit activities. Test pit subsequently filled. No samples taken for chemical analysis. Due to shallow groundwater table, filling in of test pit ___ resulted in a very soft and wet area. A mound was developed and the area roped off.
9. Express carrier picked up samples (see Sample Logbook, pages 42 through 45) at 17:50 hours. Site activities terminated at 18:22 hours. All personnel off site, gate locked.

____

Field Operations Leader

ATTACHMENT B
SAMPLE LABEL

Tetra Tech NUS, Inc. 661 Andersen Drive Pittsburgh, 15220 (412)921-7090	Project: Site: Location:			
Sample No:			Matrix:	
Date:	Time:	Preserve:		
Analysis:				
Sampled by:		Laboratory:		

ATTACHMENT C
CHAIN-OF-CUSTODY RECORD FORM

TETRA TECH NUS, INC. CHAIN OF CUSTODY | NUMBER 3413 | PAGE ____ OF _____

PROJECT NO:	FACILITY:	PROJECT MANAGER	PHONE NUMBER	LABORATORY NAME AND CONTACT:
SAMPLERS (SIGNATURE)		FIELD OPERATIONS LEADER	PHONE NUMBER	ADDRESS
		CARRIER/WAYBILL NUMBER		CITY, STATE

STANDARD TAT ☐
RUSH TAT ☐
☐ 24 hr. ☐ 48 hr. ☐ 72 hr. ☐ 7 day ☐ 14 day

CONTAINER TYPE PLASTIC (P) or GLASS (G)

PRESERVATIVE USED

TYPE OF ANALYSIS

DATE YEAR ____	TIME	SAMPLE ID	LOCATION ID	TOP DEPTH (FT)	BOTTOM DEPTH (FT)	MATRIX (GW, SO, SW, SD, QC, ETC.)	COLLECTION METHOD GRAP (G) COMP (C)	No. OF CONTAINERS									COMMENTS

1. RELINQUISHED BY	DATE	TIME	1. RECEIVED BY	DATE	TIME
2. RELINQUISHED BY	DATE	TIME	2. RECEIVED BY	DATE	TIME
3. RELINQUISHED BY	DATE	TIME	3. RECEIVED BY	DATE	TIME

COMMENTS

DISTRIBUTION: WHITE (ACCOMPANIES SAMPLE) YELLOW (FIELD COPY) PINK (FILE COPY)

4/02R
FORM NO. TtNUS-001

ATTACHMENT D
CHAIN-OF-CUSTODY SEAL

Signature

Date

CUSTODY SEAL

CUSTODY SEAL

Date

Signature

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ATTACHMENTS

1.0 PURPOSE

Decontamination is the process of removing and/or neutralizing site contaminants that have contacted and/or accumulated on equipment. The purpose of this Standard Operating Procedure (SOP) is to protect site personnel, the general public, and the environment while preserving or maintaining sample integrity. It is further intended through this procedure to describe the steps necessary for proper decontamination of drilling equipment, earth-moving equipment , chemical sampling equipment and field operation and analytical equipment.

2.0 SCOPE AND APPLICABILITY

This procedure applies to all equipment used to provide access to/acquire environmental samples that may have become contaminated through direct contact with contaminated media including air, water, and soil. This equipment includes drilling and heavy equipment and chemical sampling and field analytical equipment. Where technologically and economically feasible, single-use sealed disposable equipment will be employed to minimize the potential for cross-contamination. This SOP also provides general reference information on the control of contaminated materials.

Decontamination methods and equipment requirements may differ from one project to another. General equipment items are specified in Section 6.0, but project-specific equipment must be obtained to address the project-specific decontamination procedures presented in Section 7.0 and applicable subsections.

3.0 GLOSSARY

<ins>Alconox/Liquinox</ins> - A brand of phosphate-free laboratory-grade detergent.

<ins>Decontamination Solution</ins> - A solution selected/identified in the Health and Safety Plan or Project-Specific Quality Assurance Plan. The solution is selected and employed as directed by the project chemist/health and safety professional.

<ins>Deionized Water (DI)</ins> - Tap water that has been treated by passing through a standard deionizing resin column. This water may also pass through additional filtering media to attain various levels of analyte-free status. The DI water should meet College of American Pathologists (CAP) and National Committee for Clinical Laboratory Standards (NCCLS) specifications for reagent-grade Type I water.

<ins>Potable Water</ins> - Tap water from any municipal water treatment system. Use of an untreated potable water supply is not an acceptable substitute for tap water.

<ins>Pressure Washing</ins> - Process employing a high-pressure pump and nozzle configuration to create a high-pressure spray of potable water. High-pressure spray is employed to remove solids from equipment.

<ins>Solvent</ins> – A liquid in which solid chemicals or other liquids are dissolved. The solvent of choice is pesticide-grade isopropanol. Use of other solvents (methanol, acetone, or hexane) may be required for particular projects or for a particular purpose (e.g., removal of concentrated waste) and must be justified in the project planning documents. For example, it may be necessary to use hexane when analyzing for trace levels of pesticides, PCBs, or fuels. In addition, because many of these solvents are not miscible in water, the equipment should be air dried prior to use. Solvents should not be used on PVC equipment or well construction materials.

<ins>Steam Pressure Washing</ins> - A cleaning method employing a high-pressure spray of heated potable water to remove various organic/inorganic chemicals from equipment.

4.0 RESPONSIBILITIES AND PERSONNEL QUALIFICATIONS

Project Manager - Responsible for ensuring that all field activities are conducted in accordance with approved project plan(s) requirements.

Decontamination Personnel - Individuals assigned the task of decontamination. It is the responsibility of these individuals to understand the use and application of the decontamination process and solutions as well as the monitoring of that process to ensure that it is working properly. This is accomplished through visual evaluation, monitoring instrument scanning of decontaminated items, and/or through the collection of rinsate blanks to verify contaminant removal.

Field Operations Leader (FOL) - Responsible for the implementation of project-specific planning documents. This includes on-site verification that all field activities are performed in compliance with approved SOPs or as otherwise dictated by the approved project plan(s). The FOL is also responsible for the completion and accuracy of all field documentation.

Site Safety Officer (SSO) - Exercises shared responsibility with the FOL concerning decontamination effectiveness. All equipment arriving on site (as part of the equipment inspection), leaving the site, and moving between locations is required to go through a decontamination evaluation. This is accomplished through visual examination and/or instrument screening to determine the effectiveness of the decontamination process. Improper or incomplete decontamination is sufficient to restrict equipment from entering the site, exiting the site, or moving to a new location on the site until the objectives are successfully completed.

General personnel qualifications for decontamination activities include the following:

- Occupational Safety and Health Administration (OSHA) 40-hour and applicable refresher training.
- Capability of performing field work under the expected physical and environmental (i.e., weather) conditions.
- Familiarity with appropriate decontamination procedures.

5.0 HEALTH AND SAFETY

In addition to the health and safety issues and reminders specified in subsections of this SOP, the following considerations and requirements must be observed as SOPs for field equipment decontamination activities:

- If any solvents or hazardous chemicals (e.g., isopropyl alcohol) are to be used in equipment decontamination activities, the FOL must first obtain the manufacturer's/supplier's Material Safety Data Sheet (MSDS) and assure that it is reviewed by all users (prior to its use), added to the site Hazardous Chemical Inventory, and maintained on site as part of the project Hazard Communication Program.
- Review and observe specific health and safety requirements (e.g., personal protective equipment [PPE]) specified in the project-specific health and safety plan for this activity.

6.0 EQUIPMENT LIST

- Wood for decontamination pad construction, when applicable (see Section 7.1).

- Tools for constructing decontamination pad frame, when applicable (see Section 7.1).
- Visqueen sheeting or comparable material to cover decontamination pad frame, when applicable (see Section 7.1).
- Wash/drying racks for auger flights and drill/drive rods, when applicable (see Section 7.2).
- PPE as specified in the project health and safety plan.
- Soap and water for washing and rinsing.
- Deionized water for final rinsing.
- Solvents (e.g., pesticide-grade isopropanol) for rinsing (see applicable portions of Section 7.2).
- Tubs, buckets, etc. for containerizing rinse water (see applicable portions of Section 7.2).
- Sample bottles for collecting rinsate blanks (see Section 7.2).
- Calibrated photoionization detector (PID) or flame ionization detector (FID) to monitor decontaminated equipment for organic vapors generated through the existence of residual contamination or the presence of decontamination solvent remaining after the piece was rinsed.
- Aluminum foil or clear clean plastic bag for covering cleaned equipment (see applicable portions of Section 7.2).
- Paper towels or cloths for wiping.
- Brushes, scrapers, or other hand tools useful for removing solid materials from equipment.
- Clear plastic wrap for covering or wrapping large decontaminated equipment items (see Section 7.2.2).
- Drum-moving equipment for moving filled waste drums (optional) (see Section 7.3).
- Drum labels for waste drums (see Attachment A).

7.0 PROCEDURES

The process of decontamination is accomplished through the removal of contaminants, neutralization of contaminants, or isolation of contaminants. To accomplish this activity, preparation is required including site preparation, equipment selection, and evaluation of the decontamination requirements and processes. Site contaminant types, concentrations, and media types are primary drivers in the selection of the types of decontamination and where it will be conducted. For purposes of this SOP, discussion is limited to decontamination procedures for general environmental investigations.

Decontamination processes will be performed at the location(s) specified in project-specific planning documents. Typical decontamination locations include the following:

- Temporary decontamination pads/facilities
- Sample locations
- Centralized decontamination pad/facilities

- Combination of some or all of the above

The following discussion includes general considerations for the decontamination process. Specific construction and implementation procedures will be as specified in the project-specific planning documents and/or may be as dictated by site-specific conditions as long as the intent of the requirements in the planning documents is met. This intent is to contain any residual fluids and solids generated through the decontamination process.

7.1 Decontamination Pad Design/Construction Considerations

7.1.1 Temporary Decontamination Pads

Temporary decontamination pads may be constructed at satellite locations within the site area in support of temporary work areas. These structures are generally constructed to support the decontamination of heavy equipment such as drill rigs and earth-moving equipment but can be employed for smaller articles.

The purpose of the decontamination pad is to contain wash waters and potentially contaminated soil generated during decontamination procedures. Therefore, construction of these pads should take into account the following considerations:

- Site location – The decontamination site selected should be far enough from the work site to maximize decontamination effectiveness while minimizing travel distance. The location of the decontamination site shall be selected to provide, in the judgment of the FOL or FOL designee, compliance with as many of the following characteristics as practicable:

  - Well removed from pedestrian/vehicle thoroughfares.

  - Avoidance of areas where control/custody cannot be maintained.

  - Avoidance of areas where potential releases of contaminated media or decontamination fluids may be compounded through access to storm water transport systems, streams, or other potentially sensitive areas.

  - Avoidance of potentially contaminated areas.

  - Avoidance of areas too close to the ongoing operation, where cross-contamination may occur.

  The selected decontamination site should include the following, where possible:

  - Areas where potable water and electricity are provided.

Safety Reminder

When utilizing electrical power sources, either hard-wired or portable-generated sources, ensure that:

- All power is routed through a Ground Fault Circuit Interrupter (GFCI).

- All power cords are in good condition (no physical damage), rated for the intended energy load, and designated for outdoor use.

In situations where accomplishing these elements is not possible, it will be necessary to implement a site electrical grounding program.

- Areas where support activities such as removing decontamination waters soil and sediment are possible without entering an active exclusion zone.

- Areas that offer sufficient size to carry out the specific decontamination sequence.

• Decontamination pad (decon pad) – The decon pad shall be constructed to meet the following characteristics:

  - Size – The size of the pad should be sufficient to accept the equipment to be decontaminated as well as permitting free movement around the equipment by the personnel conducting the decontamination. The size should permit these movements utilizing pressure/steam washer wands and hoses and minimizing splash due to work in close quarters.

  - Slope – An adequate slope will be constructed to permit the collection of water and potentially contaminated soil within a trough or sump constructed at one end. The collection point for wash waters should be of adequate distance that the decontamination workers do not have to walk through the wash waters while completing their tasks. Because the pad will be sloped, place a light coating of sand over the plastic to minimize potential slips and falls. See the text about liners below.

  - Sidewalls – The sidewalls shall be at least 6 inches in height (or as high as possible if 6 inches is not achievable) to provide adequate containment for wash waters and soil. If splash represents a potential problem, splash guards should be constructed to control overspray. Sidewalls may be constructed of wood, inflatables, sand bags, etc. to permit containment. Splash guards are typically wood frames with Visqueen coverings to control overspray.

  - Liner – Depending on the types of equipment and decontamination method to be used, the liner should be of sufficient thickness to provide a puncture-resistant barrier between the decontamination operation and the unprotected environment. Care should be taken to examine the surface area prior to placing the liner to remove sharp articles (sticks, stones, debris) that could puncture the liner. Liners are intended to form an impermeable barrier. The thickness may vary from a minimum recommended thickness of 10 mil to 30 mil. The desired thickness may be achieved through layering materials of lighter construction. It should be noted that various materials (rubber, polyethylene sheeting) become slippery when wet. To minimize this potential hazard associated with a sloped liner, a light coating of sand shall be applied to provide traction as necessary.

  - Wash/drying racks – Auger flights, drill/drive rods, and similar equipment require racks positioned off of the ground to permit these articles to be washed, drained, and dried while secured from falling during this process.

For decontamination of direct-push technology (DPT) equipment, the pad may be as simple as a mortar tub containing buckets of soapy water for washing and an empty bucket to capture rinse waters. Decontamination may be conducted at the rear of the rig to permit rapid tool exchange.

• Maintenance – Maintain the decontamination area by:

  - Periodically clearing the work area of standing water, soil, and debris, and coiling hoses to aid in eliminating slip, trip, and fall hazards. In addition, these articles will reduce potential backsplash and cross-contamination.

- Regularly changing the decontamination fluids to ensure proper cleaning and prevent cross-contamination.

- PPE – Periodically evaluate the condition of, and maintain the decontamination equipment, including regular cleaning of face shields and safety glasses. This is critical to ensuring the safety of decontamination personnel and the integrity of the decontamination process, and it will ensure that equipment is functioning properly.

7.1.2 Decontamination Activities at Drill Rigs/DPT Units

During subsurface sampling activities including drilling and DPT activities, decontamination of drive rods, Macro Core Samplers, split spoons, etc. is typically conducted at an area adjacent to the operation. Decontamination is generally accomplished using a soap/water wash and rinse utilizing buckets and brushes. This area requires sufficient preparation to accomplish the decontamination objectives.

Buckets shall be placed within mortar tubs or similar secondary containment tubs to prevent splash and spills from reaching unprotected environmental media. Drying racks shall be employed as directed for temporary pads to permit parts to dry and be evaluated prior to use/reuse. Methodology regarding this activity is provided in Section 7.2.

7.1.3 Decontamination Activities at Remote Sample Locations

When sampling at remote locations, sampling equipment such as trowels and pumps/tubing should be evacuated of potentially contaminated media to the extent possible. This equipment should be wrapped in plastic for transport to the temporary/centralized decontamination location for final cleaning and disposition. Flushing and cleaning of single-use equipment such as disposable trowels, tubing, and surgeon's gloves may allow disposal of this equipment after visible soil and water remnants have been removed.

7.2 Equipment Decontamination Procedures

The following represents procedures to be employed for the decontamination of equipment that may have contacted and/or accumulated contamination through site investigation activities.

7.2.1 Monitoring Well Sampling Equipment

7.2.1.1 Groundwater sampling equipment – This includes pumps inserted into monitoring wells such as bladder pumps, Whale pumps, and Redi-Flo pumps and reusable bailers, etc.

1. Evacuate to the extent possible, any purge water within the pump/bailer.

2. Scrub using soap and water and/or steam clean the outside of the pump/bailer and, if applicable, the pump tubing.

3. Insert the pump and tubing/bailer into a clean container of soapy water. Pump/run a sufficient amount of soapy water through the pump/bailer to flush out any residual well water. After the pump is flushed, circulate soapy water through the pump to ensure that the internal components are thoroughly flushed.

4. Remove the pump and tubing/bailer from the container

5. Rinse external pump components using tap water.

6. Insert the pump and tubing/bailer into a clean container of tap water. Pump/run a sufficient amount of tap water through the pump/bailer to evacuate all of the soapy water (until clear).

CAUTION
Do not rinse PE, PVC, and associated tubing with solvents –
Use the procedures defined in the project-specific planning documents. If they are not defined, contact the FOL for guidance. The solvent rinse described in Step 7 may be omitted if groundwater does not contain oil, grease, PAHs, PCBs, or other hard to remove organic materials.

7. If groundwater contains or is suspected to contain oil, grease, PAHs, PCBs, or other hard to remove organic materials, rinse the equipment to be cleaned with pesticide-grade isopropanol.

8. Pass deionized water through the hose to flush out the tap water and solvent residue as applicable.

9. Drain residual deionized water to the extent possible.

10. Allow components of the equipment to air dry.

11. For bladder pumps, disassemble the pump and wash the internal components with soap and water, then rinse with tap water, isopropanol, and deionized water and allow to dry. After the parts are dry, conduct a visual inspection and a monitoring instrument scan to ensure that potential contaminants and all decontamination solvent have been removed. Collect a rinsate blank in accordance with the project-specific planning documents to ensure that the decontamination process is functioning as intended. The typical frequency of collection for rinsate blanks is 1 per 20 field samples. In addition, wipe samples or field tests such as UV light may be used.

12. Wrap pump/bailer in aluminum foil or a clear clean plastic bag for storage.

SAFETY REMINDER
Remember when handling powered equipment to disconnect the power source and render the equipment to a zero energy state (both potential and kinetic) before opening valves, disconnecting lines, etc.

7.2.1.2 Electronic Water Level Indicators/Sounders/Tapes

During water level measurements, rinsing the extracted tape and probe with deionized water and wiping the surface of the extracted tape between locations is acceptable. However, periodic full decontamination should be conducted as follows:

1. Wash with soap and water
2. Rinse with tap water
3. Rinse with deionized water

NOTE
In situations where oil, grease, free product, other hard to remove materials are encountered, probes and exposed tapes should be washed in hot soapy water. If probes or tapes cannot be satisfactorily decontaminated (they are still stained, discolored, etc.), they should be removed from service.

7.2.1.3 Miscellaneous Equipment

Miscellaneous equipment including analytical equipment (water quality testing equipment) shall be cleaned per manufacturers' instructions. This generally includes wiping the sensor housing and rinsing with tap and deionized water.

Coolers/shipping containers employed to ship samples are received from the laboratory in a variety of conditions including marginal to extremely poor. Coolers shall be evaluated prior to use for the following:

- Structural integrity – Coolers missing handles or having breaks in the outer housing should be removed and not used. Notify the laboratory that the risk of shipping samples in the cooler(s) provided is too great and request a replacement unit.

- Cleanliness – As per protocol, only volatile organic samples are accompanied by a trip blank. If a cooler's cleanliness is in question (visibly dirty/stained) or if there are noticeable odors, the cooler should be decontaminated prior to use as follows:

  1. Wash with soap and water
  2. Rinse with tap water
  3. Dry

If these measures fail to clean the cooler to an acceptable level, remove the unit from use as a shipping container and ask the cooler provider (e.g., the analytical laboratory) to provide a replacement unit.

7.2.2 Downhole Drilling Equipment

This includes any portion of the drill rig that is over the borehole, including auger flights, drill stems, rods, and associated tooling that would extend over the borehole. The following procedure is to be employed prior to initiating the drilling/sampling activity, then between locations:

CAUTION

Exercise care when using scrapers to remove soil and debris from downhole drilling equipment. Inadvertent slips of scrapers have resulted in cuts, scrapes, and injured knuckles, so use scrapers carefully when removing soil from these items.

1. Remove loose soil using shovels, scrapers, etc.

2. Through a combination of scrubbing using soap and water and/or steam cleaning or pressure washing, remove visible dirt/soil from the equipment being decontaminated.

CAUTION

In Step 3, do not rinse PE, PVC, and associated tubing with solvents. The appropriate procedures should be defined within the project-specific planning documents. If they are not defined, contact the FOL for guidance. The solvent rinse described in Step 4 may be omitted if groundwater does not contain oil, grease, PAHs, PCBs, or other hard to remove organic materials.

3. Rinse the equipment with tap water, where applicable (steam cleaning and pressure washing incorporate rinsing as part of the process).

4. If the equipment has directly or indirectly contacted contaminated sample media and is known or suspected of being contaminated with oil, grease, PAHs, PCBs, or other hard to remove organic materials, rinse equipment with pesticide-grade isopropanol

5. To the extent possible, allow components to air dry.

6. If the decontaminated equipment is to be used immediately after decontamination, screen it with a calibrated photoionization detector (PID)/flame ionization detector (FID) to ensure that all contaminants and possible decontamination solvents (if they were used) have been adequately removed.

7. Wrap or cover equipment in clear plastic until it is time to be used.

SAFETY REMINDER

Even when equipment is disconnected from power sources, dangers such as the following may persist:

Falls - An auger flight standing on its end may fall and injure someone. Secure all loose articles to prevent heavy articles from falling onto people or equipment.

Burns - Steam cleaner water is heated to more than 212 °F and exhibits thermal energy that can cause burns. Prevent contact of skin with hot water or surfaces.

High water pressure - Pressure washer discharge can have 2,000 to 4,000 psi of water pressure. Water under this amount of pressure can rupture skin and other human tissues. Water at 4,000 psi exiting a 0° tip can be dangerous because of its relatively high cutting power. The exit velocity and cutting power of the water are reduced when exiting a 40° fan tip, but damage to soft tissues is still possible.

In general, follow the rules below to avoid injury, equipment damage, or incomplete decontamination:

1. Read the operating manual and follow the manufacturers' recommended safety practices before operating pressure washers and steam cleaners.

2. Never point the pressure washer or steam cleaner at another person or use to clean your boots or other parts of your body. Water lacerations and burns may appear to be minor at first but can be life threatening. Do not attempt to hold small parts in your hand while washing them with high-temperature or high-pressure water.

3. Always wear PPE as specified in the HASP such as:

   - Hard hat, safety glasses, splash shield, impermeable apron or splash suit, and hearing protection. Remember that excessive noise is a hazard when operating gas-powered engines and electrically driven pressure washers. PPE will be identified in your project specific planning documents.

4. Inspect each device before use. An inspection checklist will be provided in the project-specific planning documents. If it is a rented device, safety measures are typically provided by the vendor. In all cases, if you are not familiar with the operation of a pressure washer/steam cleaner, do not operate it until you obtain and thoroughly review operating instructions and recommended safety practices.

5. Do not modify equipment unless the manufacturer has approved the modifications.

7.2.3 Soil/Sediment Sampling Equipment

This section applies to soil sampling equipment including but not limited to hand augers, stainless steel trowels/spoons, bowls, dredges, scoops, split spoons, Macro Core samplers, etc.

1. Remove all loose soil from the equipment through manual means.

2. Through a combination of scrubbing using soap and water and/or steam cleaning or pressure washing, remove visible dirt/soil from the equipment.

3. Rinse the equipment with tap water.

> ***CAUTION***
> Do not rinse PE, PVC, and associated tubing with solvents. The appropriate procedures should be defined within the project-specific planning documents. If they are not defined, contact the FOL for guidance. The solvent rinse described in Step 4 may be omitted if groundwater does not contain oil, grease, PAHs, PCBs, or other hard to remove organic materials.

4. If the equipment is contaminated or suspected to be contaminated with oil, grease, PAHs, PCBs, or other hard to remove organic materials, rinse the equipment with pesticide-grade isopropanol.

5. Rinse the equipment with deionized water.

6. To the extent possible, allow components to air dry.

7. If the equipment is to be used immediately after decontamination, screen it with a calibrated PID/FID to ensure that all solvents (if they were used) and trace contaminants have been adequately removed.

8. After the equipment has dried, wrap it in aluminum foil for storage until use.

Dredges employed in sediment sampling are typically decontaminated as follows:

- Remove the sediment sample from the sampling device

- If sufficient associated surface water is available at the sampling site, place the dredge in the water and flush to remove visible sediment.

- Extract the dredge and wash it in soap and water per the project-specific planning documents.

> ***CAUTION***
> When handling dredges, the primary safety concern is trapping fingers or extremities in the larger dredge samplers within the jaws or pinch points of the mechanical jaws. Keep hands, fingers, and extremities away from these pinch and compression points. Either handle the device by the rope or preferably lock the jaws in place to control the potential for closing during maintenance and/or cleaning.

7.3 Contact Waste/Materials

During the course of field investigations, disposable/single-use equipment becomes contaminated. These items include tubing, trowels, PPE (gloves, overboots, splash suits, etc.), and broken sample containers.

With the exception of the broken glass, single-use articles should be cleaned (washed and rinsed) of visible materials and disposed as normal refuse. The exception to this rule is that extremely soiled materials that cannot be cleaned shall be containerized for disposal in accordance with applicable federal, state, and local regulations.

7.3.1 Investigation-Derived Wastes - Decontamination Wash Waters and Sediments

NOTE
Requirements for waste storage may differ from one facility to the next. Facility-specific directions for waste storage areas will be provided in project-specific documents, or separate direction will be provided by the Project Manager.

1. Assume that all investigation-derived waste (IDW) generated from decontamination activities contains the hazardous chemicals associated with the site unless there are analytical or other data to the contrary. Waste solution volumes could vary from a few gallons to several hundred gallons in cases where large equipment required cleaning.

2. Where possible, use filtering systems to extend the use of water within a closed system wash unit to recycle water and to reduce possible waste amounts.

NOTE
Containerized waste rinse solutions are best stored in 55-gallon drums (or equivalent containers) that can be sealed until ultimate disposal at an approved facility.

3. Label waste storage containers appropriately labeled (see Attachment A).

4. Ensure that the IDW storage area is configured to meet the following specifications to permit access to the containers and to conduct spill/leak monitoring, sampling, and extraction when the disposal route is determined:

   - Enclose areas accessible by the general public using construction fencing and signs.

   - Stored materials in 55-gallon drums on pallets with four (or fewer) drums per pallet.

   - Maintain the retaining bolt and label on the outside of storage containers where readily visible.

   - Provide at least 4 feet of room between each row of pallets to allow access to containers for sampling, drum removal, and spill response.

   - As directed in project-specific planning documents, maintain an IDW Inventory List and provide the list to the site Point of Contact at the termination of each shift.

   - Maintain spill response equipment at the IDW storage area in case it is required for immediate access.

- Where possible, use equipment for moving containers. Where not possible, obtain help to manipulate containers.

CAUTION

Each container of water can weigh up to 490 pounds. Each 55-gallon drum of wet soil can weigh more than 750 pounds. Fill drums and temporary containers to 80 percent capacity to minimize spill and handling difficulties. Use drum carts to move filled drums.

See safe lifting techniques provided in Section 4.4 of the Tetra Tech NUS, Inc. Health and Safety Guidance Manual.

When placing drums, keep your fingers out of pinch and smash points such as between the drums. In some cases such as well development and/or purge water, you can place the drums to be filled on the pallet and transport materials in smaller easier to handle containers.

7.4 Decontamination Evaluation

Upon decontamination of equipment, determine the effectiveness of the decontamination process in the following manner:

- Visual evaluation – A visual evaluation will be conducted to ensure the removal of particulate matter. This shall be done to ensure that the washing/rinsing process is working as intended.

- Instrument Screening – A properly calibrated PID/FID should be used to evaluate the presence of site contaminants and solvents used in the cleaning process. The air intake of the instrument shall be passed over the article to be evaluated. Avoid placing the instrument probe into residual waters. A PID/FID reading greater than the daily established background level requires a repeat of the decontamination process, followed by rescreening with the PID/FID. This sequence must be repeated until no instrument readings greater than the daily established background level are observed. It should be noted that the instrument scan is only viable if the contaminants are detectable within the instrument's capabilities.

NOTE

When required by project-specific planning documents, collection of rinsate blanks (see next step) shall be completed without exception unless approval to not collect these samples is obtained from the Project Manager.

- Collection of Rinsate Blanks – It is recommended that rinsate samples be collected to:

  - Evaluate the decontamination procedure representing different equipment applications (pumps versus drilling equipment) and different decontamination applications.

  - Single-use disposable equipment – The number of samples should represent different types of equipment as well as different lot numbers of single-use articles.

  - The collection and the frequency of collection of rinsate samples are as follows unless specified differently in the project-specific planning documents:

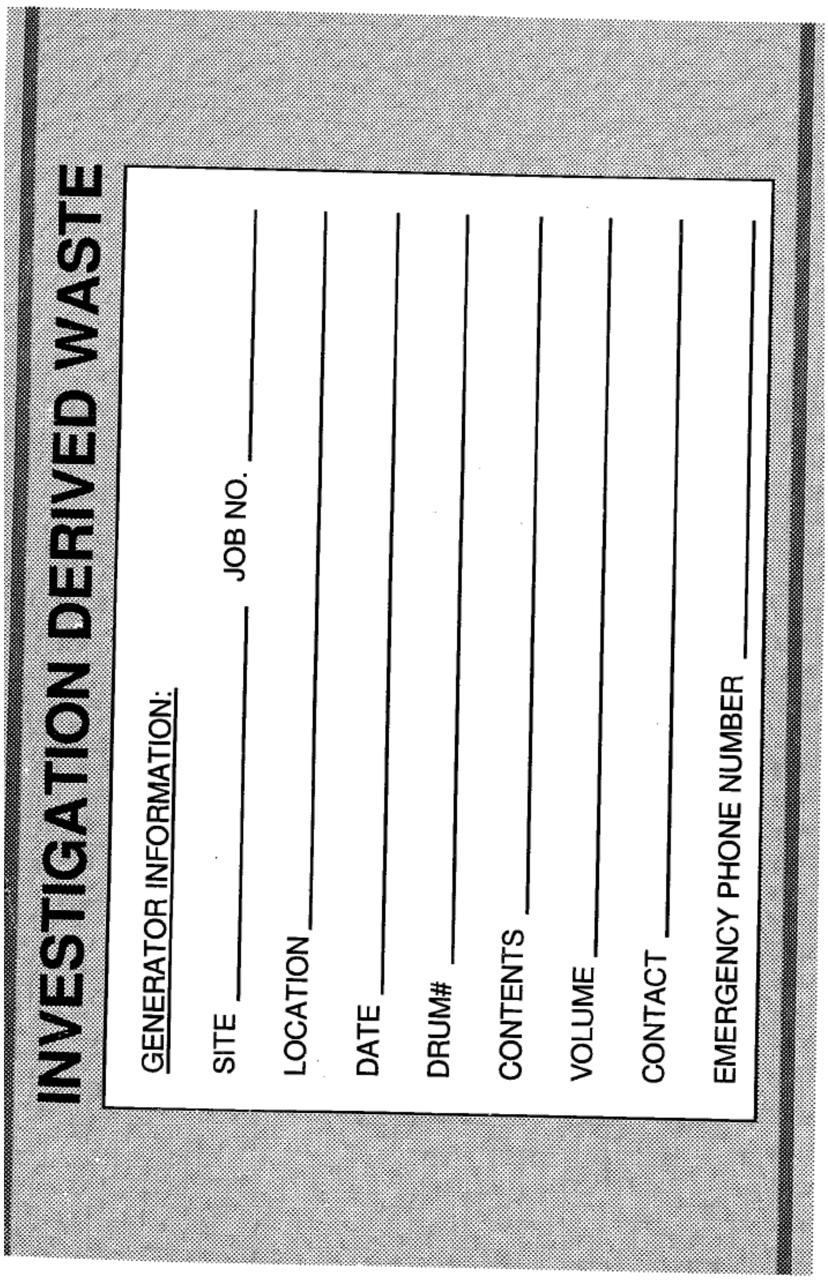
    - Per decontamination method
    - Per disposable article/batch number of disposable articles

NOTE

It is recommended that an initial rinsate sample be collected early in the project to ensure that the decontamination process is functioning properly and to avoid using a contaminated batch of single-use articles. It is recommended that a follow-up sample be collected later during the execution of the project to ensure that those conditions do not change.

Rinsate samples collection may be driven by types of and/or levels of contaminant. Difficult to remove contaminants, oils/greases, some PAHs/PCBs, etc. may also support the collection of additional rinsates due to the obvious challenges to the decontamination process. This is a field consideration to be determined by the FOL.

Attachment A
iDW Label


INVESTIGATION DERIVED WASTE

GENERATOR INFORMATION:

SITE ____________ JOB NO. ____________

LOCATION ____________

DATE ____________

DRUM# ____________

CONTENTS ____________

VOLUME ____________

CONTACT ____________

EMERGENCY PHONE NUMBER ____________

STANDARD OPERATING PROCEDURE
MUNITIONS RESPONSE PROGRAM (MRP) SOP 05
GPS DATA COLLECTION AND TRANSFER

1.0 OVERVIEW

The primary purpose of this Standard Operating Procedure (SOP) is to provide the Field Technicians with basic instructions for operating a handheld Global Positioning System (GPS) unit allowing them to set GPS parameters in the receiver, record GPS positions on the field device, and transfer the data for integration into existing Geographic Information System (GIS) figures.

This SOP is specific to GIS quality data collection for Trimble-specific hardware and software.

If possible, the Trimble GeoXT or XH Operators Manual should be downloaded onto the operator's personal computer for reference before or while in the field. The manual can be downloaded at the following website:
http://trl.trimble.com/docushare/dsweb/Get/Document-311749/TerraSyncReferenceManual.pdf

Unless the operator is proficient in the setup and operation of the GPS unit, the Project Manager (or designee) should have the GPS unit shipped to the project-specific contact listed below in the Pittsburgh, Pennsylvania office at least five working days prior to field mobilization so project-specific data files (i.e. shape files), background images, data dictionaries, and correct coordinate systems can be uploaded into the unit.

Tetra Tech NUS
Attn: Ralph Basinski
661 Anderson Drive, Bldg #7
Pittsburgh, PA 15220

The SOP also describes how field collected data is to be transferred through the use of the MRP Website. (http://www.ttnus.com/MRPRepository/). This website serves as a centralized portal to facilitate data exchange for field personnel, GIS staff, and project managers. The website contains a "Reference" page that will contain the latest version of this SOP and other valuable documentation.

For technical questions regarding operation of the GPS units and data collection, please contact John Wright (john.wright@tetratech.com). For general questions about this SOP and use of the MRP website, please contact Mark Maguire (mark.maguire@tetratech.com).

2.0 REQUIRED EQUIPMENT

The following hardware and software should be utilized for locating and establishing GPS points in the field:

2.1 GPS Hardware & Equipment

- Hand-held GPS Unit capable of sub-meter accuracy. This includes the docking cradle, a/c adapter, stylus, and USB cable for data transfer. Two models, the GeoXH and GeoXT, are acceptable for use. The XH yields higher accuracy (in both real-time and post-processed) and **<u>should always be requested</u>** when highly precise data is required.

- An external antenna will yield better satellite reception, especially in heavy tree canopy. Associated accessories include a range pole and hardware clamp, for mounting the GPS unit to the pole.

- Indelible marker.

- Non-metallic pin flags for temporary marking of positions.

2.2 GPS Software

The following software is required to transfer data from the handheld GPS unit to a personal computer:

- Trimble TerraSync version 2.6 or later (pre-loaded onto GPS unit from vendor)

- Microsoft ActiveSync version 4.5 or later. Download to personal computer from:
  http://www.microsoft.com/windowsmobile/en-us/downloads/microsoft/activesync-download.mspx

  Note: Windows Vista and Windows 7 users should download Windows Mobile Device Center version 6.1 or later from the following site, if it is not already loaded on the machine:
  http://www.microsoft.com/windowsmobile/en-us/downloads/microsoft/device-center-download.mspx

- Trimble Data Transfer Utility (<u>freeware version 2.1 or later</u>). Download to personal computer from:
  http://www.trimble.com/datatransfer.shtml

3.0 START-UP PROCEDURES

Prior to utilizing the GPS in the field, ensure the unit is fully charged. The unit may come charged from the vendor, but an overnight charge is recommended prior to fieldwork.

The Geo-series GPS units require a docking cradle for both charging and data transfer. The Geo-series GPS unit is docked in the cradle by first inserting the far domed end in the top of the cradled, then gently seating the contact end into the latch. The power charger is then connected to the cradle at the back end using the twist-lock connector. Attach a USB cable as needed between the cradle (B end) and the laptop/PC (A end).

It is recommended that the user also be familiar and check various Windows Mobile settings. One critical setting is the Power Options. The backlight should be set as needed to conserve power when not in use.

3.1 Initial Start Up

1) Power on the GPS unit by pushing the small green button located on the lower right front of the unit.

2) Utilizing the stylus that came with the GPS unit, launch **TerraSync** from the Windows Operating System by tapping on the start icon located in the upper left hand corner of the screen and then tap on **TerraSync** from the drop-down list.

3) If the unit does not default to the Setup screen, tap the Main Menu (uppermost left tab, just below the Windows icon) and select Setup.

4) If the unit was previously shipped to the Pittsburgh office for setup, you can skip directly to Section 4.0. However, to confirm or change settings, continue on to Section 3.1.

3.2 Confirm Setup Settings

Use the Setup section to confirm the TerraSync software settings. To open the Setup section, tap the Main Menu and select Setup. (Note that if the unit was shipped from the Pittsburgh office, these settings should have been set for your specific project. Feel free to contact Pittsburgh staff with any questions.)

1) Tap on the Coordinate System.
2) Verify the project specs are correct for your specific project by scrolling through the various settings. Edit as needed and then tap OK; otherwise, tap Cancel to return to Setup Menu. **Note:** It is always best to utilize the Cancel tab rather than the OK tab if no changes are made since configurations are easily changed by mistake.
3) Tap on the Units.
4) Verify the user preferences are correct for your specific project by scrolling through the various settings. Edit as needed and then tap OK; otherwise, tap Cancel to return to Setup Menu.
5) Tap Real-time Settings.

6) Verify the Real-time Settings are correct for your specific project by scrolling through the various settings. Edit as needed and then tap OK; otherwise, tap Cancel to return to Setup Menu.
7) The GPS unit is now configured correctly for your specific project.

3.3 Antenna Connection

1) If a connection has been properly made with the internal antenna, a satellite icon along with the number of usable satellites will appear at the top of the screen next to the battery icon. If no connection is made (e.g.: no satellite icon), tap on the GPS tab to connect antenna.
2) At this point the GPS unit is ready to begin collecting data.

3.4 Loading a Background file

This section provides instructions on pulling in a pre-loaded background file. These files are helpful in visualizing your current location.

1) From the Main Menu select Map, then tap on Layers, select the background file from drop down list.
2) Select the project-specific background file from the list of available files.
3) Once the selected background file appears, the operator can manipulate the screen utilizing the +/- and <-/-> functions at the bottom of the screen.
4) In operating mode, the operator's location will show up on the background file as a floating "x".

4.0 FIELD DATA COLLECTION

For MRP data collection activities, a new GPS file should be created **every day** and transferred **nightly** using the MRP website (see Section 9.0). This is to insure the timely transfer of data, file organization in the database, and allow for next-day GIS mapping. Also, individual GPS data files should be **unique to a particular site** or unit (typically a UXO number). If multiple sites are visited in a single data, multiple files should be created.

4.1 Creating a Data File

1) From the Main Menu select Data.
2) From the Sub Menu (located below the Data tab) select New which will bring up the New Data File menu.
3) An auto-generated filename appears and should be edited for your specific project. The following naming convention should be followed as closely as possible: **IH-UXO4-01012010-TeamA**, where "IH" is the installation abbreviation (Indian Head), "UXO04" is the site, and "01012010" is the data in MMDDYYYY format. If multiple teams are being deployed across an individual site on the same day, it is important to specify the

field team name at the end of the file name (“TeamA”). If the integral keyboard does not appear, tap the small keyboard icon at the bottom of the screen.

4) Select the data dictionary that will be used to collect features. The data dictionary provides predefined fields and drop-down menus to facilitate data collection as it relates to specific MRP data types. The MRP data dictionary is entitled “**MRP Data Collection**” and should appear in the data dictionary drop-down list. This should have been pre-loaded into the GPS prior to use. The data dictionary file is available on the MRP website under the “Reference” section.
5) After entering the file name and selecting the data dictionary, tap Create to create the new file.
6) Confirm antenna height if screen appears. Antenna height is the height that the GPS unit will be held from the ground surface (Typically 3 to 4 feet)
7) The Choose Feature screen appears.

4.2 Collecting Features

1) If not already open, the Collect Feature screen can be opened by tapping the Main Menu and selecting Data. The Sub Menu should default to Collect.
2) **<u>Do not</u> begin the data logging process until you are at the specific location for which you intend to log the data.**
3) A known reference or two should be shot at the beginning and at the end of each day in which the GPS unit is being used. This allows for greater accuracy during post-processing of the data.
4) Upon arriving at the specific location, select the proper feature type from the data dictionary list (MEP Object, Transect End Point, GPS QC Point, or General Point).
5) Tap Create to begin data logging.
6) As the GPS is collecting positions, enter the feature attributes, starting with the Item ID. This field is required and will not allow the user to continue or save the position without entering a value. Enter any additional notes or feature descriptions in the appropriate fields.
7) Data logging can be confirmed by viewing the writing pencil icon in the upper part of the screen. Also, the logging counter will begin. As a Rule of Thumb, accumulate a minimum of 20 readings on the counter, per point, as indicated by the logging counter before saving the GPS data.
8) Once the counter has reached a minimum number of counts (i.e. 20), tap on OK to save the data point to the GPS unit. Confirm the feature. All data points are automatically saved within the GPS unit.
9) Repeat steps 2 through 8, giving each data point a unique name or number.

Note: If the small satellite icon or the pencil icon is blinking, this is an indication the GPS unit is not collecting data. A possible problem may be too few satellites. While still in data collection mode, tap on Main Menu in upper left hand corner of the screen and select Status. Skyplot will display as the default showing the number of available satellites. To increase productivity (number of usable satellites) use the stylus to move the pointer on the productivity and precision line to the left. This will decrease precision, but increase productivity. The precision and productivity of the GPS unit can be adjusted as the number of usable satellites changes throughout the day. To determine if GPS is correctly recording data, see Section 5.2. If the precision toggle is decreased, the user should frequently check the Skyplot display to restore the default values as soon as possible.

4.3 Navigation

This section provides instructions on navigating to saved data points in an existing file within the GPS unit.

1) From the Main Menu select Map.
2) Using the Select tool, pick the point on the map to where you want to navigate.
3) The location you select will have a box placed around the point.
4) From the Options menu, choose the Set Nav Target (aka set navigation target).
5) The location will now have double blue flags indicating this point is you navigation target.
6) From the Main Menu select Navigation.
7) The dial and data on this page will indicate what distance and direction you need to travel to reach the desired target.
8) Follow the navigation guide until you reach the point you select.
9) Repeat as needed for any map point by going back to Step 1.

4.4 Data Quality Control

Quality control checks should be performed each day of data collection and/or data navigation. QC checks are important both to understand real-time accuracy while in the field, and also to provide control data needed during post-processing.

1) Known survey benchmarks, surveyed monitoring wells, or other established and documented control points should be identified
2) GPS equipment should be placed on known control points and positions recorded
3) For data collection tasks - QC check data should be collected at least at the start and completion of the fieldwork for the day of data collection. Additional occupation and collection of control point data should occur as possible during the work day, and should increase in frequency as the number of data points increase and the need for accurate data collection increases
4) For navigation tasks such as stake placement for planned sample locations, QC data checks should be done at least at the start and completion of the fieldwork for each day. Known visible targets should be occupied and observed by the user, while the GPS satellite status and other user interface data is reviewed. The user should assess whether the real-time accuracy settings on the GPS are within the tolerance of the observed visual reference points.

4.5 Viewing Data or Entering Additional Data Points to the Current File

1) To view the stored data points in the current file, tap on the Main Menu and select Map. Stored data points for that particular file will appear. Use the +/- and <-/-> icons in lower left hand corner of screen to zoom in/out and to manipulate current view.
2) To return to data collection, tap on the Main Menu and select Data. You are now ready to continue to collect additional data points.

4.6 Viewing Data or Entering Data Points from an Existing File

1) To view data points from a previous file, tap on Main Menu and select Data, then select File Manager from the Sub Menu.
2) Highlight the file you want to view and select Map from the Main Menu.
3) To add data points to this file, tap on Main Menu and select Data. Continue to collect additional data points.

4.7 Shutting Down

This section provides instruction for properly shutting down the GPS unit.

1) When shutting down the GPS unit for the day, first click on the "X" in the upper right hand corner.
2) You will be prompted to ensure you want to exit TerraSync. Select Yes.
3) Power off the GPS unit by pushing the small green button located on the bottom face of the unit.
4) Place the GPS unit in its cradle to recharge the battery overnight. Ensure the green charge light is visible on the charging cradle.

5.0 DATA TRANSFER

This section describes how data should be downloaded from the GPS units and uploaded to a central website for post-processing and integration into GIS datasets. GPS data collected on a given day should be transferred **that night** for post-processing by GIS staff the next morning. Once post-processed, the GPS data will be plotted on a map and be immediately provided to the project team for review. Data upload, download, and review will be facilitated through a secure MRP website: http://www.ttnus.com/MRPRepository/

5.1 Load Data from the GPS Unit to Your Computer

1) Install the Data Transfer and ActiveSync software installed on your PC (see section 2.2)
2) Connect the GeoXH/XT to your PC via an A/B USB cable (blade end and square end type "HP printer" style)
3) ActiveSync should auto-detect the connection and recognize the data collector
4) Make sure the data file desired is CLOSED in TerraSync prior to transfer
5) Connect via ActiveSync as a guest (not a partnership)

6) Run the Trimble Data Transfer Utility program on your PC
7) Select "***GIS Datalogger on Windows CE***" or similar selection
8) Hit the green connect icon to the right - the far right area should say "***Connected to ....***" if successful
9) Select the "***Receive***" data tab (under device)
10) Select "***Data***" from file types on the right
11) Find the file(s) needed for data transfer. You can sort the data files by clicking on the date/time header
12) Select or browse to a C-drive folder you can put this file for upload
13) When the file appears on the list, hit the "***Transfer All***". Once complete, a packet of multiple data files will appear on your computer in the specified folder.

5.2 Gain Access to MRP Website

1) Confirm that your computer has internet access
2) Click on the following link: http://www.ttnus.com/MRPRepository/
3) To register for the website, click on the "Register here" link. Enter your information and click "Submit." NOTE: Requests for registration are sent to Ralph Basinski, Program Manager, for approval. Please contact mark.maguire@tetratech.com if you experience any access issues.
4) Enter your username (Tetra Tech email address) and password to log in.

5.3 Upload GPS Data from Your Computer to the MRP Website

1) From the main page, select "Upload" from the menu at left.
2) Select the type of data you are uploading, typically "GPS Field Data"
3) Select the appropriate Installation and Site. Remember that GPS files should be unique for each site, even if multiple sites are visited in one day. If collected data is not associated with a site, select "Other."
4) Select "browse" to navigate to the appropriate *.SSF file on your computer. When you use the Trimble download utility to grab data from the GPS unit, multiple files will appear on your computer. You only need to the upload the *.SSF file.
5) Populate the "Comments" field to describe the dataset and any other pertinent information. This information will be provided to the GIS analyst who will be integrating the dataset, so be sure to be as descriptive as possible especially if there are any issues with the data. (For example, if you were to sample 16 points and for some reason you believe only 15 were logged, it is helpful to share this information.)
7) Select "Upload." Users will be notified if the files were uploaded successfully.

5.4 Download Data from the MRP Website to Your Computer

The download utility on the MRP website will serve different user types. **Field staff** will use the utility to download GIS figures (in PDF format) and view the previous day(s) field data on aerial photographs, checking for any discrepancies or missing data elements. **Project Managers** will also have the ability to download and view these figures, to visualize the data and track project progress. This utility will also allow **GIS Analysts** to download the *.SSF files posted by field staff for post-processing and map plotting.

To download GIS Figures:

1) From the main page, select "Download" from the menu at left.
2) Select an Installation and Site
3) Users can view Figures for a particular date or by a range of dates, by selecting the ` appropriate options. <u>To search all dates, leave all of these fields as the default</u>.
4) Select "Search"
5) A table will appear showing the files available for download. Simply click on the link to the file and you will be prompted to save it to your computer.

FC 1000. CLEANING / DECONTAMINATION PROCEDURES

1. PERFORMANCE CRITERIA

1.1. The cleaning/decontamination procedures must ensure that all equipment that contacts a sample during sample collection is free from the analytes of interest and constituents that would interfere with the analytes of interest.

1.2. The detergents and other cleaning supplies cannot contribute analytes of interest or interfering constituents unless these are effectively removed during a subsequent step in the cleaning procedure.

1.3. The effectiveness of any cleaning procedure (including all cleaning reagents) must be supported by equipment blanks with reported non-detected values.

The cleaning procedures outlined in this SOP are designed to meet the above-mentioned performance criteria. Alternative cleaning reagents or procedures may be used. However, the organization must be prepared to demonstrate through documentation (i.e., company-written protocols and analytical records) and historical data (i.e., absence of analytes of interest in equipment blanks) that it consistently meets these performance criteria. Field quality control measures (see FQ 1210) must support the use of alternative reagents or procedures.

FC 1001. *Cleaning Reagents*

Recommendations for the types and grades of various cleaning supplies are outlined below. The recommended reagent types or grades were selected to ensure that the cleaned equipment is free from any detectable contamination.

1. DETERGENTS: Use Luminox (or a non-phosphate solvent based equivalent), Liqui-Nox (or a non-phosphate equivalent) or Alconox (or equivalent). EPA recommends Luminox (or equivalent) since solvent rinses can be eliminated from the cleaning process. Liquinox (or equivalent) may be substituted (solvent rinses, when applicable, must be performed), and Alconox (or equivalent) may be substituted if the sampling equipment will not be used to collect phosphorus or phosphorus-containing compounds.

2. SOLVENTS

Note: If the detergent Luminox (or equivalent) is used, solvent rinses are not required.

2.1. Use pesticide grade isopropanol as the rinse solvent in routine equipment cleaning procedures. This grade of alcohol must be purchased from a laboratory supply vendor.

2.2. Other solvents, such as acetone or methanol, may be used as the final rinse solvent if they are pesticide grade. However, methanol is more toxic to the environment and acetone may be an analyte of interest for volatile organics.

2.2.1. **Do not use** acetone if volatile organics are of interest.

2.3. Properly dispose of all wastes according to applicable regulations. Containerize all solvents (including rinsates) for on-site remediation or off-site disposal, as required.

2.4. Pre-clean equipment that is heavily contaminated (see FC 1120, section 3) with organic analytes with reagent grade acetone and hexane or other suitable solvents.

2.5. Use pesticide grade methylene chloride when cleaning sample containers.

2.6. Store all solvents away from potential sources of contamination (gas, copier supplies, etc.).

3. ANALYTE-FREE WATER SOURCES

3.1. Analyte-free water is water in which all analytes of interest and all interferences are below method detection limits.

3.2. Maintain documentation (such as results from equipment blanks) to demonstrate the reliability and purity of analyte-free water source(s).

3.3. The source of the water must meet the requirements of the analytical method and must be free from the analytes of interest. In general, the following water types are associated with specific analyte groups:

- Milli-Q (or equivalent polished water): suitable for all analyses.
- Organic-free: suitable for volatile and extractable organics.
- Deionized water: not suitable for volatile and extractable organics if the analytes of interest are present in concentrations that affect the result.
- Distilled water: not suitable for volatile and extractable organics, metals or ultra-trace metals.

3.4. Use analyte-free water for blank preparation and the final decontamination water rinse.

3.5. In order to minimize long-term storage and potential leaching problems, obtain or purchase analyte-free water just prior to the sampling event. If obtained from a source (such as a laboratory), fill the transport containers and use the contents for a single sampling event. Empty the transport container(s) at the end of the sampling event.

3.6. Discard any analyte-free water that is transferred to a dispensing container (such as a wash bottle) at the end of each sampling day.

4. ACIDS

4.1. <u>Reagent Grade Nitric Acid</u>: 10 - 15% (one volume concentrated nitric acid and five volumes deionized water).

4.1.1. Use for the acid rinse unless nitrogen components (e.g., nitrate, nitrite, etc.) are to be sampled.

4.1.2. If sampling for ultra-trace levels of metals, use an ultra-pure grade acid.

4.2. <u>Reagent Grade Hydrochloric Acid</u>: 10% hydrochloric acid (one volume concentrated hydrochloric and three volumes deionized water).

4.2.1. Use when nitrogen components are to be sampled.

4.3. If samples for both metals and the nitrogen-containing components (see FC 1001, section 4.1.1 above) are collected with the equipment, use the hydrochloric acid rinse, or thoroughly rinse with hydrochloric acid after a nitric acid rinse.

4.4. If sampling for ultra trace levels of metals, use an ultra-pure grade acid.

4.5. Freshly prepared acid solutions may be recycled during the sampling event or cleaning process. Dispose appropriately at the end of the sampling event, cleaning process or if acid is discolored or appears otherwise contaminated (e.g., floating particulates).

4.5.1. Transport only the quantity necessary to complete the sampling event.

4.6. Dispose of any unused acids according to FDEP and local ordinances.

FC 1002. *Reagent Storage Containers*

The contents of all containers must be clearly marked.

1. DETERGENTS: Store in the original container or in a high density polyethylene (HDPE) or polypropylene (PP) container.

2. SOLVENTS

   2.1. Store solvents to be used for cleaning or decontamination in the original container until use in the field. If transferred to another container for field use, the container must be either glass or Teflon.

   2.2. Use dispensing containers constructed of glass, Teflon, or stainless steel. Note: if stainless steel sprayers are used, any components (including gaskets and transfer lines) that contact the solvents must be constructed of inert materials.

3. ANALYTE-FREE WATER: Transport in containers appropriate to the type of water to be stored. If the water is commercially purchased (e.g., grocery store), use the original containers when transporting the water to the field. Containers made of glass, Teflon, polypropylene, or Polyethylene (PE) are acceptable.

   3.1. Use glass, Teflon, polypropylene or PE to transport organic-free sources of water on-site.

   3.2. Dispense water from containers made of glass, Teflon, PE or polypropylene.

   3.3. Do not store water in transport containers for more than three days before beginning a sampling event.

   3.4. Store and dispense acids using containers made of glass, Teflon, PE or polypropylene.

FC 1003. *General Requirements*

1. Before using any equipment, clean/decontaminate all sampling equipment (pumps, tubing, lanyards, split spoons, etc.) that are exposed to the sample.

   1.1. Before installing, clean (or obtain as certified precleaned) all equipment that is dedicated to a single sampling point and remains in contact with the sample medium (e.g., permanently installed groundwater pump (see FS 2220, section 3.3.4).

   1.2. Clean this equipment any time it is removed for maintenance or repair.

   1.3. Replace dedicated tubing if discolored or damaged.

2. Clean all equipment in a designated area having a controlled environment (house, laboratory, or base of field operations) and transport to the field precleaned and ready to use, unless otherwise justified.

3. Rinse all equipment with water after use, even if it is to be field-cleaned for other sites. Rinse equipment used at contaminated sites or used to collect in-process (e.g., untreated or partially treated wastewater) samples immediately with water.

4. Whenever possible, transport sufficient clean equipment to the field so that an entire sampling event can be conducted without the need for cleaning equipment in the field.

5. Segregate equipment that is only used once (i.e., not cleaned in the field) from clean equipment and return to the in-house cleaning facility to be cleaned in a controlled environment.

6. Protect decontaminated field equipment (including well sounders) from environmental contamination by securely wrapping and sealing with one of the following:

   6.1. Aluminum foil (commercial grade is acceptable);

   6.2. Untreated butcher paper; or

   6.3. Clean, untreated, disposable plastic bags. Plastic bags may be used:

   - For all analyte groups except volatile and extractable organics;
   - For volatile and extractable organics, if the equipment is first wrapped in foil or butcher paper or if the equipment is completely dry.

7. Containerize all solvent rinsing wastes, detergent wastes and other chemical wastes requiring off-site or regulated disposal. Dispose of all wastes in conformance with applicable regulations.

FC 1100. Cleaning Sample Collection Equipment

FC 1110. ON-SITE/IN-FIELD CLEANING

1. Cleaning equipment on-site is not recommended because:

   1.1. Environmental conditions cannot be controlled.

   1.2. Wastes (solvents and acids) must be containerized for proper disposal.

2. If performed, follow the appropriate cleaning procedure as outlined in FC 1130. Ambient temperature water may be substituted in the hot, sudsy water bath, and hot water rinses.

Note: Properly dispose of all solvents and acids.

3. Rinse all equipment with water after use, even if it is to be field-cleaned for other sites. Rinse equipment used at contaminated sites or used to collect in-process (e.g., untreated or partially treated wastewater) samples immediately with water.

FC 1120. HEAVILY CONTAMINATED EQUIPMENT

In order to avoid contaminating other samples, isolate heavily contaminated equipment from other equipment and thoroughly decontaminate the equipment before further use. Equipment is considered heavily contaminated if it:

- Has been used to collect samples from a source known to contain significantly higher levels than background;
- Has been used to collect free product; or
- Has been used to collect industrial products (e.g., pesticides or solvents) or their by-products.

1. Cleaning heavily contaminated equipment in the field is not recommended.

2. ON-SITE PROCEDURES

   2.1. Protect all other equipment, personnel and samples from exposure by isolating the equipment immediately after use.

2.2. At a minimum, place the equipment in a tightly sealed untreated plastic bag.

2.3. Do not store or ship the contaminated equipment next to clean, decontaminated equipment, unused sample containers, or filled sample containers.

2.4. Transport the equipment back to the base of operations for thorough decontamination.

2.5. If cleaning must occur in the field, and in order to document the effectiveness of the procedure, collect and analyze blanks on the cleaned equipment (see FQ 1000).

3. CLEANING PROCEDURES

3.1. If organic contamination cannot be readily removed with scrubbing and a detergent solution, prerinse equipment by thoroughly rinsing or soaking the equipment in acetone.

3.1.1. Do not use solvent soaks or rinses if the material is clear acrylic.

3.1.2. Use hexane only if preceded and followed by acetone.

3.2. In extreme cases, it may be necessary to steam clean the field equipment before proceeding with routine cleaning procedures.

3.3. After the solvent rinses (and/or steam cleaning), use the appropriate cleaning procedure (see FC 1130).

3.3.1. Scrub, rather than soak all equipment with sudsy water.

3.3.2. If high levels of metals are suspected and the equipment cannot be cleaned without acid rinsing, soak the equipment in the appropriate acid. Do not use stainless steel equipment when heavy metal contamination is suspected or present, since stainless steel cannot be exposed to prolonged acid soaks.

3.4. If the field equipment cannot be cleaned utilizing these procedures, discard unless further cleaning with stronger solvents and/or oxidizing solutions is effective as evidenced by visual observation and blanks.

3.5. Clearly mark or disable all discarded equipment to discourage use.

FC 1130. GENERAL CLEANING

Follow these procedures when cleaning equipment under controlled conditions. See FC 1110 for modifications if cleaning is performed on-site. <u>Check manufacturer's instructions for cleaning restrictions and/or recommendations</u>.

FC 1131. *Procedure for Teflon, Stainless Steel and Glass Sampling Equipment*

This procedure must be used when sampling for **ALL** analyte groups: extractable organics, metals, nutrients, etc. or if a single decontamination protocol is desired to clean all Teflon, stainless steel and glass equipment.

1. Rinse equipment with hot tap water.

2. Soak equipment in a hot, sudsy water solution (Liqui-Nox or equivalent - see FC 1001, section 1).

3. If necessary, use a brush to remove particulate matter or surface film.

4. Rinse thoroughly with hot tap water.

5. If samples for trace metals or inorganic analytes will be collected with the equipment and the equipment **is not** stainless steel, thoroughly rinse (wet all surfaces) with the appropriate acid solution (see FC 1001, section 4).

6. Rinse thoroughly with analyte-free water. Use enough water to ensure that all equipment surfaces are thoroughly flushed with water.

7. If samples for volatile or extractable organics will be collected, rinse with isopropanol. Wet equipment surfaces thoroughly with free-flowing solvent. Rinse thoroughly with analyte-free water (see FC 1001, section 3).

8. Allow to air dry. Wrap and seal according to FC 1003, section 6 as soon as the equipment is air-dried.

9. If isopropanol is used, the equipment may be air-dried without the final analyte-free water rinse (see FC 1131, section 8 above); however, **the equipment must be completely dry before wrapping or use**.

10. Wrap clean sampling equipment according to the procedure described in FC 1003, section 6.

FC 1132. *General Cleaning Procedure for Plastic Sampling Equipment*

1. Rinse equipment with hot tap water.

2. Soak equipment in a hot, sudsy water solution (Liqui-Nox or equivalent - see FC 1001, section 1).

3. If necessary, use a brush to remove particulate matter or surface film.

4. Rinse thoroughly with hot tap water.

5. Thoroughly rinse (wet all surfaces) with the appropriate acid solution (see FC 1001, section 4). Check manufacturer's instructions for cleaning restrictions and/or recommendations.

6. Rinse thoroughly with analyte-free water. Use enough water to ensure that all equipment surfaces are thoroughly flushed with water. Allow to air dry as long as possible.

7. Wrap clean sampling equipment according to the procedure described in FC 1003, section 6.

FC 1133. *Cleaning Procedure by Analyte Group*

See Table FC 1000-1 for the procedures to be used to decontaminate equipment based on construction of sampling equipment, and analyte groups to be sampled.

FC 1140. AUTOMATIC SAMPLERS, SAMPLING TRAINS AND BOTTLES

1. When automatic samplers are deployed for extended time periods, clean the sampler using the following procedures when routine maintenance is performed. Inspect deployed samplers prior to each use. At a minimum, change the tubing if it has become discolored or has lost elasticity (FC 1140, section 2.3 below).

2. Clean all automatic samplers (such as ISCO) as follows:

   2.1. Wash the exterior and accessible interior portions of the automatic samplers (excluding the waterproof timing mechanisms) with laboratory detergent (see FC 1001, section 1) and rinse with tap water.

2.2. Clean the face of the timing case mechanisms with a clean, damp cloth.

2.3. Check all tubing (sample intake and pump tubing). Change the tubing every six months (if used frequently) or if it has become discolored (i.e., affected by mold and algae) or if it has lost its elasticity.

2.4. See FC 1160, section 4 for the procedures associated with cleaning the tubing in the pump head.

3. AUTOMATIC SAMPLER ROTARY FUNNEL AND DISTRIBUTOR

3.1. Clean with hot sudsy water and a brush (see FC 1001, section 1 for appropriate detergent type).

3.2. Rinse thoroughly with analyte-free water.

3.3. Air dry.

3.4. Replace in sampler.

4. SAMPLER METAL TUBE: Clean as outlined in FC 1160, section 5.

5. REUSABLE GLASS COMPOSITE SAMPLE CONTAINERS

5.1. If containers are used to collect samples that contain oil, grease or other hard to remove materials, it may be necessary to rinse the container several times with reagent-grade acetone before the detergent wash. If material cannot be removed with acetone, discard the container.

5.2. Wash containers following the procedure outlined in FC 1131 above. End with a final solvent rinse if organics are to be sampled.

5.3. Invert containers to drain and air dry for at least 24 hours.

5.4. Cap with aluminum foil, Teflon film or the decontaminated Teflon-lined lid.

5.5. After use, rinse with water in the field, seal with aluminum foil to keep the interior of the container wet, and return to the laboratory or base of operations.

5.6. **Do not recycle or reuse containers if**:

5.6.1. They were used to collect in-process (i.e., untreated or partially treated) wastewater samples at industrial facilities;

5.6.2. A visible film, scale or discoloration remains in the container after the cleaning procedures have been used; or

5.6.3. The containers were used to collect samples at pesticide, herbicide or other chemical manufacturing facilities that produce toxic or noxious compounds. Such containers must be properly disposed of (preferably at the facility) at the conclusion of the sampling activities.

5.6.4. If the containers described above are reused, check no less than 10% of the cleaned containers for the analytes of interest **before** use. If found to be contaminated, (i.e., constituents of interest are found at method detection levels or higher), then **discard the containers**.

6. REUSABLE PLASTIC COMPOSITE SAMPLE CONTAINERS

6.1. Follow FC 1132.

6.2. Inspect the containers. Determine if the containers can be reused by the criteria in FC 1140, section 5 above.

7. GLASS SEQUENTIAL SAMPLE BOTTLES FOR AUTOMATIC SAMPLER BASED FOR SEQUENTIAL MODE

7.1. Clean glass sequential sample bottles to be used for collecting inorganic samples by using a laboratory dishwasher (see FC 1140, sections 7.1.1 through 7.1.3 below) or manually following the procedures in FC 1131.

7.1.1. Rinse with appropriate acid solution (see FC 1001, section 4).

7.1.2. Rinse thoroughly with tap water.

7.1.3. Wash in dishwasher at wash cycle, using laboratory detergent cycle, followed by tap and analyte-free water rinse cycles.

7.2. Replace bottles in covered, automatic sampler base; cover with aluminum foil for storage.

7.3. Rinse bottles in the field with water as soon as possible after sampling event.

8. Glass Sequential Sample Bottles (Automatic Sampler based for Sequential Mode) to be used for Collecting Samples for Organic Compounds

8.1. Use cleaning procedures outlined in FC 1131. Allow containers to thoroughly air dry before use.

8.2. Replace bottles in covered, automatic sampler base; cover with aluminum foil for storage.

9. BOTTLE SIPHONS USED TO TRANSFER SAMPLES FROM COMPOSITE CONTAINERS

9.1. Rinse tubing with solvent and dry overnight in a drying oven.

9.2. Cap ends with aluminum foil and/or Teflon film for storage.

9.3. Seal in plastic for storage and transport.

9.4. Flush siphon thoroughly with sample before use.

10. REUSABLE TEFLON COMPOSITE MIXER RODS

10.1. Follow procedures outlined in FC 1131.

10.2. Wrap in aluminum foil for storage.

FC 1150. FILTRATION EQUIPMENT

1. Dissolved Constituents using in-line, Molded and Disposable Filter Units

1.1. Peristaltic Pump

1.1.1. Clean the pump following procedures in FC 1170, section 2.2.

1.1.2. Clean the pump head tubing following FC 1160, section 4.

1.1.3. If Teflon tubing is used, clean following the procedures in FC 1160, section 3.

1.1.4. Clean other tubing types such as polyethylene according to the appropriate procedures listed in FC 1160, section 7.

1.2. Other Equipment Types (e.g., pressurized Teflon bailer)

1.2.1. Follow the appropriate cleaning regimen specified in FC 1131 through FC 1132 for other types of equipment that utilize in-line, molded and disposable filters.

2. Dissolved Constituents using Non-disposable Filtration Units (e.g., syringes, "tripod assembly")

2.1. Stainless Steel or Glass Units

2.1.1. Follow FC 1131, assembling and applying pressure to the apparatus after each rinse step (water and acid) to drive rinsing solution through the porous filter holder in the bottom of the apparatus.

2.1.2. Remove and clean any transfer tubing according to the appropriate cleaning procedures (see FC 1160).

2.1.3. Assemble the unit and cap both the pressure inlet and sample discharge lines (or whole unit if a syringe) with aluminum foil to prevent contamination during storage.

2.1.4. If the unit will **not** be used to filter volatile or extractable organics, seal the unit in an untreated plastic bag to prevent contamination.

2.2. Reusable In-Line Filter Holders

2.2.1. Clean, using FC 1131, (if Teflon, glass or stainless steel) or FC 1132 (if plastic) assembling and applying pressure to the apparatus after each rinse step (water and acid) to drive rinsing solution through the porous filter holder in the bottom of the apparatus.

2.2.2. Assemble the unit and wrap with aluminum foil to prevent contamination during storage.

2.2.3. If the unit will **not** be used to filter volatile or extractable organics, seal the unit in an untreated plastic bag to prevent contamination.

3. FILTERS

3.1. Do not clean filters. Instructions for rinsing the filters prior to use are discussed in the applicable sampling SOPs (FS 2000 - FS 8000).

FC 1160. SAMPLE TUBING DECONTAMINATION

1. Check tubing:

1.1. For discoloration: Remove discolored tubing from use until it can be cleaned. If the discoloration cannot be removed, discard the tubing.

1.2. For elasticity (if used in a peristaltic-type pump): Discard any tubing that has lost its elasticity.

2. Transport all tubing to the field in precut, **precleaned** sections.

3. TEFLON, POLYETHYLENE AND POLYPROPYLENE TUBING

3.1. New Tubing: Follow this procedure unless the manufacturer/supplier provides certification that the tubing is clean.

3.1.1. Teflon

3.1.1.1. Rinse outside of tubing with pesticide-grade solvent (see FC 1001, section 2).

3.1.1.2. Flush inside of tubing with pesticide-grade solvent.

3.1.1.3. Dry overnight in drying oven or equivalent (zero air, nitrogen, etc.).

3.1.2. Polyethylene and Polypropylene

3.1.2.1. Clean the exterior and interior of the tubing by soaking in hot, sudsy water.

3.1.2.2. Thoroughly rinse the exterior and interior of the tubing with tap water, followed by analyte-free water.

3.2. Reused Tubing

Use the following procedure for in-lab cleaning. **Field cleaning is not recommended**:

3.2.1. Clean the exterior of the tubing by soaking in hot, sudsy water (see FC 1001, section 1) in a stainless steel sink (or equivalent non-contaminating material). Use a brush to remove any particulates, if necessary.

3.2.2. Use a small bottle brush and clean the inside of the tubing ends where the barbs are to be inserted or cut 1-2 inches from the ends of the tubing after cleaning.

3.2.3. Rinse tubing exterior and ends liberally with tap water.

3.2.4. Rinse tubing surfaces and ends with the appropriate acid solution (see FC 1001, section 4), tap water, isopropanol (see FC 1001, section 2), and finally analyte-free water.

3.2.4.1. Note: Eliminate the isopropanol rinse for polyethylene or polypropylene tubing.

3.2.5. Place tubing on fresh aluminum foil or clean polyethylene sheeting. Connect all of the precut lengths of tubing with Teflon inserts or barbs.

3.2.6. Cleaning configuration:

3.2.6.1. Place cleaning reagents: [sudsy water (see FC 1001, section 1); acid (see FC 1001, section 4); isopropanol (see FC 1001, section 2)] in an appropriately cleaned container (2-liter glass jar is recommended).

3.2.6.2. Place one end of the Teflon tubing into the cleaning solution.

3.2.6.3. Attach the other end of the Teflon tubing set to the influent end of a pump.

3.2.6.4. Recycle the effluent from the pump by connecting a length of Teflon tubing from the effluent to the glass jar with the cleaning reagents.

3.2.6.5. Recycling as described above may be done for all reagents listed in FC 1160, section 3.2.6.1 above, **except** the final isopropanol rinse and the final analyte-free water rinse. Disconnect the tubing between the effluent end of the pump and the jar of cleaning reagents.

3.2.6.6. Containerize isopropanol in a waste container for proper disposal.

3.2.6.7. Analyte-free water may be discarded down the drain.

3.2.7. Using the above configuration described in FS 1160, section 3.2.6 above:

3.2.7.1. Pump hot, sudsy water through the connected lengths. Allow the pump to run long enough to pump at least three complete tubing volumes through the tubing set.

3.2.7.2. Using the same procedure, successively pump tap water, the acid solution(s), tap water, isopropanol, and finally analyte-free water through the system.

3.2.7.3. Leave the Teflon inserts or barbs between the precut lengths and cap or connect the remaining ends.

3.2.8. After the interior has been cleaned as described in FC 1160, section 3.2.7 above, rinse the exterior of the tubing with analyte-free water.

3.2.9. Wrap the connected lengths in aluminum foil or untreated butcher paper and store in a clean, dry area until use.

4. Flexible Tubing used in Pump Heads of Automatic Samplers and other Peristaltic Pumps

Replace tubing after each sampling point if samples are collected through the tubing. Unless the pump is deployed to collect samples from the same location over a long period of time, remove and wash the tubing after each sampling event (see FC 1140, section 1).

4.1. Flush tubing with hot tap water then sudsy water (see FC 1001, section 1).

4.2. Rinse thoroughly with hot tap water.

4.3. Rinse thoroughly with analyte-free water.

4.4. If used to collect metals samples, flush the tubing with an appropriate acid solution (see FC 1001, section 4), followed by thorough rinsing with analyte-free water. If used to collect both metals and nitrogen components use hydrochloric acid (see FC 1001, section 4.1.1).

4.5. Install tubing in peristaltic pump or automatic sampler.

4.6. Cap both ends with aluminum foil or equivalent.

Note: Change tubing at specified frequencies as part of routine preventative maintenance.

5. STAINLESS STEEL TUBING

Clean the exterior and interior of stainless steel tubing as follows:

5.1. Using sudsy water (see FC 1001, section 1), scrub the interior and exterior surfaces.

5.2. Rinse with hot tap water.

5.3. Rinse with analyte-free water.

5.4. If volatile or extractable organics are to be sampled, rinse all surfaces with isopropanol (see FC 1001, section 2). Use enough solvent to wet all surfaces with free flowing solvent.

5.5. Allow to air dry or thoroughly rinse with analyte-free water.

6. GLASS TUBING

6.1. Use new glass tubing.

6.2. If volatile or extractable organics are to be sampled, rinse with isopropanol (see FC 1001, section 2).

6.3. Air dry for at least 24 hours.

6.4. Wrap in aluminum foil or untreated butcher paper to prevent contamination during storage.

6.5. Discard tubing after use.

7. MISCELLANEOUS NON-INERT TUBING TYPES (TYGON, RUBBER, PVC, ETC.)

7.1. New Tubing

7.1.1. As a general rule, new tubing may be used without preliminary cleaning.

7.1.2. Protect new tubing from potential environmental contamination by wrapping in aluminum foil and sealing in untreated plastic bags or keep in the original sealed packaging until use.

7.1.3. If new tubing is exposed to potential contamination, rinse the exterior and interior tubing surfaces with hot tap water followed by a thorough rinse with analyte-free water.

7.1.4. If new tubing is to be used to collect samples, thoroughly rinse the tubing with sample water (i.e., pump sample water through the tubing) before collecting samples.

7.2. Reused Tubing

7.2.1. Flush tubing with sudsy solution of hot tap water and laboratory detergent (see FC 1001, section 1).

7.2.2. Rinse exterior and interior thoroughly with hot tap water.

7.2.3. Rinse exterior and interior thoroughly with analyte-free water.

7.2.4. If used to collect only metals samples, flush the tubing with nitric acid (see FC 1001, section 4.1), followed by a thorough rinse with analyte-free water.

7.2.5. If used to collect metals and nitrogen-containing compounds, see FC 1001, section 4.3.

7.2.6. Cap ends in aluminum foil and store in clean, untreated plastic bags to prevent contamination during storage and transport.

FC 1170. PUMPS

1. SUBMERSIBLE PUMPS

1.1. Pumps used for Purging and Sampling Metals and/or Volatile and Extractable Organics

1.1.1. Construction of pump body and internal mechanisms (bladders, impellers, etc.), including seals and connections, must follow Tables FS 1000-1, FS 1000-2 and FS 1000-3.

1.1.2. Tubing material must follow Tables FS 1000-1, FS 1000-2 and FS 1000-3.

1.1.3. Clean pump exterior following FC 1132. Note: omit the solvent rinse if the pump body is constructed of plastic (e.g., ABS, PVC, etc.).

1.1.4. Clean the pump internal cavity and mechanism as follows:

1.1.4.1. If used only for purging, thoroughly flush the pump with water before purging the next well.

1.1.4.2. When used for purging and sampling, completely disassemble the pump (if practical) and decontaminate between each well.

1.1.4.3. When used for purging and sampling and the pump cannot be (practicably) disassembled, then clean the internal cavity/mechanism by pumping several gallons of sudsy water (see FC 1001, section 1), followed by several gallons of tap water, and finally, several gallons of analyte-free water.

1.1.4.4. If multiple sampling points are located in an area that is not accessible by a vehicle, and it is difficult to return to the vehicle for cleaning or to transport all cleaning materials to the staging location, at a minimum thoroughly rinse the pump with water.

1.1.5. Refer to FC 1160, section 3 to clean Teflon tubing.

1.1.6. Refer to FC 1160, section 5 for stainless steel tubing.

1.1.7. Clean other types of tubing according to FC 1160, sections 6 and 7.

1.2. <u>Pumps used for Purging and Sampling all Analytes except Metals, Volatile and Extractable Organics</u>

1.2.1. Pump construction: no restrictions.

1.2.2. Pump tubing material: no restrictions.

1.2.3. Scrub the exterior of the pump with appropriate metal-free, phosphate-free or ammonia-free detergent solution.

1.2.4. Rinse the exterior with tap water and analyte-free water.

1.2.5. Rinse the interior of the pump and tubing by pumping tap or analyte-free water through the system using a clean bucket or drum.

2. ABOVE-GROUND PUMPS USED FOR PURGING AND SAMPLING

2.1. <u>Pumps used only for Purging</u>

2.1.1. The exterior of the pump must be free of oil and grease.

2.1.2. Select tubing according to Tables FS 1000-1, FS 1000-2 and FS 1000-3.

2.1.3. Clean the tubing that contacts the formation water according to the appropriate protocol for construction materials specified in FC 1160.

2.2. <u>Pumps used for Sampling</u>

2.2.1. Clean the exterior of the pump with a detergent solution followed by a tap water rinse. Use clean cloths or unbleached paper towels that have been moistened with the appropriate solution to wipe down the pump.

2.2.2. Select tubing according to Tables FS 1000-1, FS 1000-2 and FS 1000-3.

2.2.3. Clean the tubing that contacts the formation water according to the appropriate protocol for construction materials specified in FC 1160.

FC 1180. ANALYTE-FREE WATER CONTAINERS

This section pertains to containers that are purchased to transport, store and dispense analyte-free water. It does not apply to water that has been purchased in containers. See FC 1002, section 3 for appropriate construction materials.

1. NEW CONTAINERS

1.1. Wash containers and caps according to FC 1131, omitting the solvent rinse if plastic (polyethylene or polypropylene) containers are being cleaned.

1.2. Cap with Teflon film or the bottle cap. The bottle cap must be composed of the same material as the container and cannot be lined.

2. REUSED CONTAINERS

2.1. Immediately after emptying, cap with aluminum foil, Teflon film or the container cap.

2.2. Wash the exterior of the container with lab-grade detergent solution (see FC 1001, section 1) and rinse with analyte-free water.

2.3. Rinse the interior thoroughly with analyte-free water.

2.4. Invert and allow to drain and dry.

FC 1190. ICE CHESTS AND SHIPPING CONTAINERS

1. Wash the exterior and interior of all ice chests with laboratory detergent (see FC 1001, section 1) after each use.

2. Rinse with tap water and air dry before storing.

3. If the ice chest becomes severely contaminated with concentrated waste or other toxic or hazardous materials clean as thoroughly as possible, render unusable, and properly dispose.

FC 1200. Field Instruments and Drilling Equipment

FC 1210. FIELD INSTRUMENTS (TAPES, METERS, ETC.)

Follow manufacturer's recommendations for cleaning instruments. At a minimum:

1. Wipe down equipment body, probes, and cables with lab-grade detergent solution (see FC 1001, section 1). Check manufacturer's instructions for recommendations and/or restrictions on cleaning.

2. Rinse thoroughly with tap water.

3. Rinse thoroughly with analyte-free water.

4. Store equipment according to the manufacturer's recommendation or wrap equipment in aluminum foil, untreated butcher paper or untreated plastic bags to eliminate potential environmental contamination.

FC 1220. SOIL BORING EQUIPMENT

This section pertains only to equipment that is not used to collect samples. Clean split spoons, bucket augers and other sampling devices according to FC 1131.

1. Remove oil, grease, and hydraulic fluid from the exterior of the engine and power head, auger stems, bits and other associated equipment with a power washer or steam jenny or wash by hand with a brush and sudsy waster (no degreasers).

2. Rinse thoroughly with tap water.

FC 1230. WELL CASING CLEANING

These are recommended procedures for cleaning well casing and riser pipes. Use procedures specified by a FDEP contract, order, permit, or rule, if different or more stringent than the procedures outlined below.

1. FDEP recommends only using casing that is designed for subsurface environmental groundwater monitoring.

2. Casing that has been contaminated with grease, hydraulic fluid, petroleum fuel, etc. may require additional cleaning or deemed unusable.

3. All casings and riser pipes should be cleaned before installation, unless the casing is received wrapped and ready for installation:

   3.1. Steam clean all casings and riser pipes except PVC. Steam cleaning criteria shall meet the following: water pressure - 2500 psi; water temperature - 200°F.

   3.2. Rinse thoroughly with tap (potable) water. This tap water must be free of the analytes of interest.

FC 1300. Sample Containers

FC 1310. OBTAINING CLEAN CONTAINERS

1. Obtain clean sample containers in one of three ways:

   1.1. From commercial vendors as precleaned containers. The cleaning grades must meet EPA analyte specific requirements. Keep all records for these containers (lot numbers, certification statements, date of receipt, etc.) and document the container's intended uses;

   1.2. From internal groups within the organization that are responsible for cleaning and maintaining containers according to the procedures outlined in FC 1320; or

   1.3. From a subcontracted laboratory that is accredited under the National Environmental Laboratory Accreditation Program (NELAP).

      1.3.1. The contractor must verify that the laboratory follows the container cleaning procedures outlined in FC 1320.

      1.3.2. If the laboratory cleaning procedures are different, the contractor must require that the laboratory use the following cleaning procedures or provide documentation and historical records to show that their in-house procedure produces containers that are free from the analytes of interest.

FC 1320. CONTAINER CLEANING PROCEDURES

1. Refer to Table FC 1000-2. Follow the cleaning steps in the order specified in the chart.

2. Cleaning procedures that are different from those outlined in FC 1320 may be used as long as blanks collected in the containers are free from the analytes of interest and any analytical interferences and the cleaning procedures are supported by historical and continuing documentation.

3. Inspect all containers before cleaning.

   3.1. **Do not recycle or reuse containers if**:

      3.1.1. Containers were used to collect in-process (i.e., untreated or partially treated) wastewater samples at industrial facilities;

      3.1.2. A visible film, scale or discoloration remains in the container after the cleaning procedures have been used; or

3.1.3. Containers were used to collect samples at pesticide, herbicide or other chemical manufacturing facilities that produce toxic or noxious compounds. Such containers shall be properly disposed of (preferably at the facility) at the conclusion of the sampling activities.

3.1.4. If the containers described above are reused, check no less than 10% of the cleaned containers for the analytes of interest before use. If found to be contaminated (i.e., analytes of interest are found at MDL levels or higher), discard the containers.

FC 1400. Documentation

Document cleaning procedures described below for the indicated activities. See FD 1000 for additional information about required records and retention of documents.

FC 1410. FIELD EQUIPMENT

1. IN-FIELD CLEANING

   1.1. Initially identify the procedures that are used to clean equipment in the field by SOP numbers and dates of usage.

   1.2. Record the date and time that equipment was cleaned.

2. IN-HOUSE CLEANING

   2.1. Retain any cleaning certificates, whether from a laboratory or commercial vendor.

   2.2. Identify the procedure(s) that are used to clean equipment by the SOP number and dates of usage.

   2.3. Record the date that the equipment was cleaned.

FC 1420. SAMPLE CONTAINERS

1. Organizations that order precleaned containers must retain the packing slips, and lot numbers of each shipment, any certification statements provided by the vendor and the vendor cleaning procedures.

2. Organizations that clean containers must maintain permanent records of the following:

   2.1. Procedure(s) used to clean containers by SOP number and dates of usage.

   2.2. If containers are certified clean by the laboratory the laboratory must record:

   - Type of container;
   - Date cleaned;
   - SOP used;
   - Person responsible for cleaning;
   - Lot number (date of cleaning may be used) of the batch of containers that were cleaned using the same reagent lots and the same procedure;
   - The results of quality control tests that were run on lot numbers; and
   - Any additional cleaning or problems that were encountered with a specific lot.

FC 1430. REAGENTS AND OTHER CLEANING SUPPLIES

Maintain a record of the lot number with the inclusive dates of use for all acids, solvents, and other cleaning supplies.

Appendix FC 1000
Tables, Figures and Forms

Table FC 1000-1
Procedures for Decontamination at the Base of Operations or On-Site

Construction Material	Analyte Group Sampled	SOP Reference	Base of Operations	On-Site
Teflon or Glass	All	FC 1131	Follow as written	May substitute ambient temperature water for the hot water rinses and hot detergent solution
	Extractable & Volatile Organics Petroleum Hydrocarbons		May omit acid rinse	May substitute ambient temperature water for the hot water rinses and hot detergent solution May omit acid rinse
	Metals[1] Radionuclides For ultra trace metals, refer to FS 8200		May omit solvent rinse	May substitute ambient temperature water for the hot water rinses and hot detergent solution May omit solvent rinse
	Inorganic Nonmetallics Physical & Aggregate Properties Aggregate Organics Biologicals Volatile Inorganics		May omit solvent rinse	Rinse several times with water Rinse several times with sample water from the next sampling location
	Microbiological – Viruses Microbiological - Bacteria		Omit solvent and acid rinses	Rinse several times with water Rinse several times with sample water from the next sampling location
Metallic (stainless steel, brass, etc.)	All Extractable & Volatile Organics Petroleum Hydrocarbons	FC 1131	Omit the acid rinse	May substitute ambient temperature water for the hot water rinses and hot detergent solution Omit the acid rinse
	Metals Radionuclides		Omit the acid rinse May omit the solvent rinse	May substitute ambient temperature water for the hot water rinses and hot detergent solution Omit the acid rinse May omit the solvent rinse
	Inorganic Nonmetallics Physical & Aggregate Properties Aggregate Organics Biologicals Volatile Inorganics		Omit solvent rinse May omit the acid rinse	Rinse several times with water Rinse several times with sample water from the next sampling location

Table FC 1000-1
Procedures for Decontamination at the Base of Operations or On-Site

Construction Material	Analyte Group Sampled	SOP Reference	Base of Operations	On-Site
	Microbiological – Viruses Microbiological - Bacteria		Omit solvent and acid rinses	Rinse several times with water Rinse several times with sample water from the next sampling location
Plastic (Polyethylene, polypropylene, PVC, silicone, acrylic	Volatile and Extractable Organics;	FC 1132	Follow as written.	May substitute ambient temperature water for the hot water rinses and hot detergent solution
	Inorganic Nonmetallics Physical & Aggregate Properties Aggregate Organics Biologicals Volatile Inorganics		May omit the acid rinse	Rinse several times with water Rinse several times with sample water from the next sampling location
	Microbiological – Viruses Microbiological - Bacteria		Omit acid rinse	Rinse several times with water Rinse several times with sample water from the next sampling location

[i] Do not use glass if collecting samples for boron or silica.

Table FC 1000-2
Container Cleaning Procedures

ANALYSIS / ANALYTE GROUP	CLEANING STEPS See Description Below
Extractable Organics	1, 2, 4, 6 (not required if Luminox (or equivalent is used), (5 and 7 optional), 11
Volatile Organics	1, 2, 4, (6 optional, methanol only), 7
Metals	1, 2, 3, 4, 8, 11 ** **Procedures to clean containers for ultra-trace metals are found in FS 8200
Inorganic Nonmetallics, Radionuclides, Physical and Aggregate Properties, Aggregate Inorganics, and Volatile Inorganics	1, 2, 3*, 4, 8, 11 * For nutrients, replace nitric acid with hydrochloric acid, or use a hydrochloric acid rinse after the nitric acid rinse. See FC 1001, section 4
Petroleum Hydrocarbons, and Oil and Grease	1, 2, 3, 4, (5, 6, 7 optional), 11
Microbiological (all)	1, 2, 4, 8, 9, 11
Toxicity Tests (Includes Bioassays)	1, 2, 10, 2, 4, 6.1, (10 optional), 11

NOTE: Steps 1 and 2 may be omitted when cleaning new, uncertified containers.

1. Wash with hot tap water and a brush using a suitable laboratory-grade detergent:

    1.1. Volatile and Extractable Organics, Petroleum Hydrocarbon, Oil and Grease: Luminox, Liqui-Nox, Alconox or equivalent;

    1.2. Inorganic nonmetallics: Liqui-Nox or equivalent;

    1.3. Metals: Liqui-Nox, Acationox, Micro or equivalents:

    1.4. Microbiologicals (all): Must pass an inhibitory residue test.

2. Rinse thoroughly with hot tap water.
3. Rinse with 10% nitric acid solution.
4. Rinse thoroughly with analyte-free water (deionized or better).
5. Rinse thoroughly with pesticide-grade methylene chloride.
6. Rinse thoroughly with pesticide-grade isopropanol, acetone or methanol.

    6.1. For bioassays, use only acetone, and only when containers are glass.

7. Oven dry at 103°C to 125°C for at least 1 hour.

Table FC 1000-2
Container Cleaning Procedures

7.1. VOC vials and containers must remain in the oven in a contaminant-free environment until needed. They should be capped in a contaminant-free environment just prior to dispatch to the field.

8. Invert and air-dry in a contaminant-free environment.

9. Sterilize containers:

 9.1. Plastic: 60 min at 170°C, loosen caps to prevent distortion.

 9.2. Glass: 15 min at 121°C.

10. Rinse with 10% hydrochloric acid followed by a sodium bicarbonate solution.

11. Cap tightly and store in a contaminant-free environment until use. Do not use glass if collecting samples for boron or silica.

FD 1000. DOCUMENTATION PROCEDURES

1. INTRODUCTION:

   1.1. For the creation of clear, accurate and methodical records to document all field activities affecting sample data, implement the following standard operating procedures for sample collection, sample handling and field-testing activities.

2. SCOPE AND APPLICABILITY

   2.1. This SOP provides a detailed listing of the information required for documentation of all sampling procedures and field testing.

   2.2. Refer to the associated sampling or field testing SOP for any requirements for the chronological or sequential documentation of data.

3. QUALITY ASSURANCE

   3.1. Implement review procedures to monitor and verify accurate manual and automated data entry and recordkeeping for all documentation tasks outlined in this SOP.

FD 1100. Universal Documentation Requirements

Incorporate efficient archival design and concise documentation schemes for all record systems. Ensure that the history of a sample is clearly evident in the retained records and documentation and can be independently reconstructed.

1. CRITERIA FOR ALL DOCUMENTS

   1.1. Keep all applicable documentation available for inspection. Keep all original data and records as well as reduced or manipulated forms of the original data or records.

      1.1.1. Authorized representatives of DEP have the legal right to inspect and request copies of any records using paper, electronic media, or other media during any DEP audit of physical facilities or on-site sampling events, and for any data validations conducted for applicable project data submitted to DEP.

   1.2. Record enough information so that clarifications, interpretations, or explanations of the data are not required from the originator of the documentation.

   1.3. Clearly indicate the nature and intent of all documentation and all record entries.

   1.4. Link citations to SOPs and other documents by the complete name, reference or publication number, revision number, and revision date for the cited document, when applicable. Also assign this information to internally generated SOPs.

   1.5. Retain copies of all revisions of all cited documents as part of the documentation archives.

2. PROCEDURES

   2.1. Sign, initial or encode all documentation entries made to paper, electronic or other records with a link indicating the name and responsibility of the author making the data entry, clearly indicating the reason for the signature, initials or code (e.g., “sampled by”; “released by”; “prepared by”; “reviewed by”).

   2.2. In order to abbreviate record entries, make references to procedures written in internal SOPs or methodology and procedures promulgated by external sources.

2.2.1. Document the intent to use SOPs other than the DEP SOPs, or to use allowable modifications to the DEP SOPs by recording the effective date of use for all such SOPs or modifications.

2.2.1.1. Retain any correspondence with DEP regarding approval to use alternative procedures for any projects.

2.2.2. Authorize all internal SOPs with the signatures of the quality assurance officer(s) and manager(s) responsible for implementation of the SOPs. Record the dates of signature.

2.3. Employ straightforward archiving of records to facilitate documentation tracking and retrieval of all current and archived records for purposes of inspection, verification, and historical reconstruction of all procedures and measurement data.

2.4. Keep copies or originals of all documentation, including documentation sent to or received from external parties.

2.5. Use waterproof ink for all paper documentation.

2.6. Do not erase or obliterate entry errors on paper records. Make corrections by marking a line through the error so that it is still legible. Initial or sign the marked error and its correction.

2.7. Maintain electronic audit trails for all edited electronic records, if possible. Utilize software that allows tracking of users and data edits, if available. Software that prompts the user to double-check edits before execution is also preferred. See FD 1200.

2.8. Clearly link all documentation associated with a sample or measurement. Make cross-references to specific documentation when necessary.

2.9. Link final reports, data summaries, or other condensed versions of data to the original sample data, including those prepared by external parties.

3. RETENTION REQUIREMENTS

3.1. Per the DEP QA Rule, 62-160.220 & .340, F.A.C., keep all documentation archives for a minimum of 5 years after the date of project completion or permit cycle unless otherwise specified in a Department contract, order, permit, or Title 62 rules.

FD 1200. Electronic Documentation

Handle electronic (digital) data as with any data according to applicable provisions of FD 1100.

1. RETENTION OF AUTOMATIC DATA RECORDING PRODUCTS

1.1. For data not directly read from the instrument display and manually recorded, retain all products or outputs from automatic data recording devices, such as strip chart recorders, integrators, data loggers, field measurement devices, computers, etc. Store records in electronic, magnetic, optical, or paper form, as necessary.

1.1.1. Retain all original, raw output data. Ensure archiving of these data prior to subsequent reduction or other manipulation of the data.

1.2. Identify output records as to purpose, analysis date and time, field sample identification number, etc. Maintain clear linkage with the associated sample, other data source or measured medium and specific instrument used to make the measurement.

2. ELECTRONIC DATA SECURITY

2.1. Control levels of access to electronic data systems as required to maintain system security and to prevent unauthorized editing of data.

2.2. Do not alter raw instrumentation data or original manual data records in any fashion without retention of the original raw data.

2.3. Maintain secure computer networks and appropriate virus protection as warranted for each system design.

3. ELECTRONIC DATA STORAGE AND DOCUMENTATION

3.1. Store all electronic, magnetic, and optical media for easy retrieval of records.

3.1.1. Ensure that all records can be printed to paper if needed for audit or verification purposes.

3.1.2. If it is anticipated that the documentation archive will become unreadable due to obsolescence of a particular storage technology, retain a paper archive of the data or transfer to other suitable media.

3.2. For easy retrieval of records, link all stored data to the associated sample data or other data source.

3.3. Back up all data at a copy rate commensurate with the level of vulnerability of the data. Consider replicating all original data as soon as possible after origination.

4. SOFTWARE VERIFICATION

4.1. Ensure that any software used to perform automatic calculations conforms to required formulas or protocols.

4.2. Document all software problems and their resolution in detail, where these problems have irretrievably affected data records or linkage. Record the calendar date, time, responsible personnel, and relevant technical details of all affected data and software files. Note all software changes, updates, installations, etc. per the above concerns. File and link all associated service records supplied by vendors or other service personnel.

5. PROTECTION OF EQUIPMENT AND STORAGE MEDIA

5.1. Place stationary computers, instrumentation, and peripheral devices in locations of controlled temperature and humidity and away from areas where the potential for fluid leaks, fire, falling objects, or other hazards may exist. In the field, protect portable equipment from weather, excess heat or freezing, storage in closed vehicles, spillage from reagents and samples, etc.

5.2. Protect storage media from deteriorating conditions such as temperature, humidity, magnetic fields, or other environmental hazards as above.

6. ELECTRONIC SIGNATURES – Documents signed with electronic signatures must be consistent with the requirements of 62-160.405, F.A.C.:

6.1. the integrity of the electronic signature can be assured;

6.2. the signature is unique to the individual;

6.3. the organization using electronic signatures has written policies for the generation and use of electronic signatures; and

6.4. the organization using electronic signatures has written procedures for ensuring the security, confidentiality, integrity and auditability of each signature.

FD 1300. Documentation Using Other Media

1. UNIVERSAL REQUIREMENTS

1.1. Handle documentation prepared using other media according to FD 1100.

2. PROTECTION OF STORED MEDIA

2.1. Store media such as photographs, photographic negatives, microfilm, videotape, etc. under conditions generally prescribed for these media by manufacturers and conducive to long-term storage and protection from deterioration. See also FD 1200, section 5, above.

FD 2000. DOCUMENTATION OF CLEANED EQUIPMENT, SAMPLE CONTAINERS, REAGENTS AND SUPPLIES

When providing sample containers, preservation reagents, analyte-free water or sampling equipment, document certain aspects of these preparations.

1. EQUIPMENT CLEANING DOCUMENTATION

1.1. Document all cleaning procedures by stepwise description in an internal SOP if cleaning procedures in the DEP SOP have been modified for use. Alternatively, cite the DEP SOP procedures in the cleaning record for the applicable equipment.

1.2. Record the date of cleaning.

1.2.1. If items are cleaned in the field during sampling activities for a site, document the date and time when the affected equipment was cleaned. Link this information with the site and the cleaning location at the site.

1.3. Retain or make accessible any certificates of cleanliness issued by vendors supplying cleaned equipment or sample containers.

1.3.1. Retain from the vendor or document for internal cleaning the following information for sample containers, as applicable:

- Packing slip and cleanliness certificates from vendors
- Container types and intended uses
- Lot numbers or other designations for groups of containers cleaned together using the same reagents and procedures
- Dates of cleaning
- Cleaning procedures or reference to internal cleaning SOPs or DEP SOPs
- Cleaning personnel names
- Results of quality control analyses associated with container lots
- Comments about problems or other information associated with container lots

2. SAMPLING KIT DOCUMENTATION

If supplied to a party other than internal staff, transmit to the recipient the following information pertaining to sampling equipment or other implements, sample containers, reagent containers, analyte-free water containers, reagents or analyte-free water supplied to the recipient.

- Quantity, description and material composition of all containers, container caps or closures or liners for caps or closures
- Intended application for each sample container type indicated by approved analytical method or analyte group(s)
- Type, lot number, amount and concentration of preservative added to clean sample containers and/or shipped as additional preservative
- Intended use for any additional preservatives or reagents provided
- Description of any analyte-free water (i.e., deionized, organic-free, etc.)
- Date of analyte-free water containerization
- Date of sampling kit preparation
- Description and material composition of all reagent transfer implements (e.g., pipets) shipped in the sampling kit and the analyte groups for which the implements have been cleaned or supplied
- Quantity, description and material composition of all sampling equipment and pump tubing (including equipment supplied for filtration) and the analyte groups for which the equipment has been cleaned or supplied
- Tare weight of VOC vials, as applicable (this item is necessary when EPA 5035 VOC sample vials are provided for soil samples)

3. DOCUMENTATION FOR REAGENTS AND OTHER CHEMICALS

3.1. Keep a record of the lot numbers and inclusive dates of use for all reagents, detergents, solvents and other chemicals used for cleaning and sample preservation.

3.1.1. See FD 4000 below for documentation requirements for reagents used for field testing.

FD 3000. DOCUMENTATION OF EQUIPMENT MAINTENANCE

1. Log all maintenance and repair performed for each instrument unit, including routine cleaning procedures, corrective actions performed during calibrations or verifications, and solution or parts replacement for instrument probes.

1.1. Include the calendar date for the procedures performed.

1.2. Record names of personnel performing the maintenance or repair tasks.

1.2.1. Describe any malfunctions necessitating repair or service.

2. Designate the identity of specific instrumentation in the documentation with a unique description or code for each instrument unit employed. This identifier may include a manufacturer name, model number, serial number, inventory number, or other unique identification.

3. Retain vendor service records for all affected instruments.

4. Record the following for rented equipment:

- Rental date(s)
- Equipment type and model or inventory number or other description

5. Retain the manufacturer's operating and maintenance instructions.

FD 4000. DOCUMENTATION FOR CALIBRATION OF FIELD-TESTING INSTRUMENTS AND FIELD ANALYSES

Document acceptable instrument or measuring system calibration for each field test or analysis of a sample or other measurement medium.

FD 4100. General Documentation for all Field Testing

1. STANDARD AND REAGENT DOCUMENTATION: Document information about standards and reagents used for calibrations, verifications, and sample measurements.

    1.1. Note the date of receipt, the expiration date and the date of first use for all standards and reagents.

        1.1.1. Document acceptable verification of any standard used after its expiration date.

    1.2. Record the concentration or other value for the standard in the appropriate measurement units.

        1.2.1. Note vendor catalog number and description for preformulated solutions as well as for neat liquids and powdered standards.

        1.2.2. Retain vendor assay specifications for standards as part of the calibration record.

            1.2.2.1. Record the grade of standard or reagent used.

    1.3. When formulated in-house, document all calculations used to formulate calibration standards.

        1.3.1. Record the date of preparation for all in-house formulations.

    1.4. Describe or cite the procedure(s) used to prepare any standards in-house (DEP SOP or internal SOP).

2. FIELD INSTRUMENT CALIBRATION DOCUMENTATION: Document acceptable calibration and calibration verification for each instrument unit and field test or analysis, linking this record with affected sample measurements.

    2.1. Retain vendor certifications of all factory-calibrated instrumentation.

    2.2. Designate the identity of specific instrumentation in the documentation with a unique description or code for each instrument unit used.

        2.2.1. Record manufacturer name, model number, and identifying number such as a serial number for each instrument unit.

    2.3. Record the time and date of all initial calibrations and all calibration verifications.

    2.4. Record the instrument reading (value in appropriate measurement units) of all calibration verifications.

    2.5. Record the name of the analyst(s) performing the calibration or verification.

2.6. Document the specific standards used to calibrate or verify the instrument or field test with the following information:

- Type of standard or standard name (e.g., pH buffer)
- Value of standard, including correct units (e.g., pH = 7.0 SU)
- Link to information recorded according to section 1 above

2.7. Retain manufacturers' instrument specifications.

2.8. Document whether successful initial calibration occurred.

2.9. Document whether each calibration verification passed or failed.

2.10. Document, according to records requirements of FD 3000, any corrective actions taken to modify instrument performance.

2.10.1. Document date and time of any corrective actions.

2.10.2. Note any incidence of discontinuation of use of the instrument due to calibration failure.

2.11. Describe or cite the specific calibration or verification procedure performed (DEP SOP or internal SOP).

3. Record all field-testing measurement data, to include the following:
    - Project name
    - Date and time of measurement or test (including time zone, if applicable)
    - Source and location of the measurement or test sample (e.g., monitoring well identification number, outfall number, station number or other description)
    - Latitude and longitude of sampling source location (if required)
    - Analyte or parameter measured
    - Measurement or test sample value
    - "J" data qualifier code for estimated measurement or test sample value
    - Reporting units for the measurement
    - Initials or name of analyst performing the measurement
    - Unique identification of the specific instrument unit used for the test (see 2.2 above)

FD 5000. DOCUMENTATION OF SAMPLE COLLECTION, PRESERVATION AND TRANSPORT

Follow these procedures for all samples. See FD 5100 - FD 5427 below for additional documentation for specific sampling activities. See example Forms in FD 9000 below for example formats for documenting specific sampling and testing procedures.

1. SAMPLE IDENTIFICATION REQUIREMENTS

1.1. Ensure that labels are waterproof and will not disintegrate or detach from the sample container when wet, especially under conditions of extended submersion in ice water typically accumulating in ice chests or other transport containers.

1.2. Label or tag each sample container with a unique field identification code that adequately distinguishes each sample according to the following criteria. The code must adequately link the sample container with all of the information about the sample contained in the permanent field record.

1.2.1. Link the unique field identification code to the sample source or sampling point identification, the date of sample collection, the time of sample collection (for maximum holding times equal to or less than 48 hours), the analytes of interest and the preservation technique.

1.2.2. Label or tag each sample container for the following types of samples with a unique field identification code:

- Quality control samples such as duplicate samples, other replicate samples or split samples collected for the same analyte or group of analytes
- Field samples or quality control samples collected using a different sample collection technique for the same analyte or group of analytes (for example, if both a bailer and a pump are used to collect samples for metals analysis, label the bailer sample to distinguish it from the pump sample)

1.2.3. The color, size, shape, or material composition of sample containers and caps cannot substitute for the information required in 1.2.1. – 1.2.2. Above.

1.2.4. The unique field identification code and any other information included on the container label or tag must allow the analyzing laboratory to independently determine the sample collection date, the sample collection time (for maximum holding times $\leq$ 48 hours), the sample preservation and the analytical tests to be performed on each container or group of containers.

1.3. Attach the label or tag so that it does not contact any portion of the sample that is removed or poured from the container.

1.4. Record the unique field identification code on all other documentation associated with the specific sample container or group of containers.

2. GENERAL REQUIREMENTS FOR SAMPLING DOCUMENTATION: Record the following information for all sampling:

2.1. Names of all sampling team personnel on site during sampling

2.2. Date and time of sample collection (indicate hours and minutes)

2.2.1. Use 24-hour clock time or indicate A.M. and P.M.

2.2.2. Note the exact time of collection for individual sample containers for time-sensitive analyses with a maximum holding time of 48 hours of less.

2.3. Ambient field conditions, to include, but not limited to information such as weather, tides, etc.

2.4. Comments about samples or conditions associated with the sample source (e.g., turbidity, sulfide odor, insufficient amount of sample collected)

2.5. Specific description of sample location, including site name and address

2.5.1. Describe the specific sampling point (e.g., monitoring well identification number, outfall number, station number, etc.).

2.5.2. Determine latitude and longitude of sampling source location (if required).

2.5.3. Locate sampling points on scaled maps or drawings where applicable.

2.6. Record the unique field identification code for each sample container and parameters to be analyzed, per section 1 above. The code must adequately link the sample container or group of containers with all of the information about the sample contained in the permanent field record.

2.7. Number of containers collected for each unique field identification code

2.8. Matrix sampled

2.9. Type of field sample collected, such as grab, composite or other applicable designation.

2.10. Field-testing measurement data:

2.10.1. See FD 4000 above for specific details.

2.11. Calibration records for field-testing equipment

2.11.1. See FD 4000 above for specific details.

2.12. Preservation for each container

2.12.1. Indicate whether samples are chemically preserved on-site by the sampling team or, alternatively, were collected in prepreserved (predosed) containers.

2.12.2. Indication of any tests performed in the field to determine the presence of analytical interferences in the sample.

2.12.3. Indication of any treatments of samples performed in the field to eliminate or minimize analytical interferences in the sample.

2.12.4. See FD 5100, section 1.

2.13. Purging and sampling equipment used, including the material composition of the equipment and any expendable items such as tubing.

2.14. Types, number, collection location and collection sequence of quality control samples

2.14.1. Include a list of equipment that was rinsed to collect any equipment blanks.

2.15. Use of fuel powered vehicles and equipment

2.16. Number of subsamples and amount of each subsample in any composite samples

2.16.1. Include sufficient location information for the composite subsamples per 2.4 above.

2.17. Depth of all samples or subsamples

2.18. Signature(s) or initials of sampler(s)

3. SAMPLE TRANSMITTAL RECORDS: Transmit the following information to the analytical laboratory or other receiving party. Link transmittal records with a given project and retain all transmittal records.

- Site name and address – Note: Client code is acceptable if samples are considered sensitive information and if the field records clearly trace the code to a specified site and address.
- Date and time of sample collection

- Name of sampler responsible for sample transmittal
- Unique field identification codes for each sample container
- Total number of samples
- Required analyses
- Preservation protocol
- Comments about sample or sample conditions
- Identification of common carrier (if used)

4. SAMPLE TRANSPORT

4.1. If shipping transmittal forms in the transport containers with the samples, place the forms in a waterproof enclosure and seal.

4.2. For common carrier shipping, seal transport containers securely with strapping tape or other means to prevent lids from accidentally opening.

4.2.1. Keep all shipping bills from common carriers with archived transmittal records.

5. ANCILLARY FIELD RECORDS: Link any miscellaneous or ancillary records (photographs, videotapes, maps, etc.) to specific sampling events such that these records are easily traceable in the data archives associated with the project, sampling date and sample source(s).

FD 5100. Documentation Specific To Aqueous Chemistry Sampling

1. SAMPLE PRESERVATION: Document preservation of all samples according to the following instructions.

1.1. List the chemical preservatives added to the sample.

1.2. Record the results of pH verification performed in the field, including the pH value of the sample (if applicable). Note any observations about changes in the sample as a result of adding preservative to the sample or mixing the sample with the preservative.

1.3. Record the amount of preservative added to samples and the amount of any additional preservative added. The amount dosed into sample containers supplied with premeasured preservatives must also be recorded.

1.3.1. For documentation of procedures for preservation for routine samples, cite DEP SOPs or internal SOPs for this information.

1.3.2. Record instances of deviation from preservation protocols found in SOPs when non-routine or problematic samples are collected.

1.4. Record the use of ice or other cooling method, when applicable.

2. GROUNDWATER SAMPLING

2.1. Record or establish a documentation link to the following information for all samples. See section 3 below for in-place plumbing:

- Well casing composition and diameter of well casing
- A description of the process and the data used to design the well

- The equipment and procedure used to install the well
- The well development procedure
- Pertinent lithologic or hydrogeologic information
- Ambient conditions at the wellhead or sampling point that are potential sources of unrepresentative sample contamination
- Water table depth and well depth
- Calculations used to determine purge volume
- Total amount of water purged
- Date well was purged
- Purging equipment used
- Sampling equipment used
- Well diameter
- Total depth of well
- Depth to groundwater
- Volume of water in the well
- Purging method
- Placement depth of tubing or pump intake
- Depth and length of screened interval
- Times for beginning and ending of purging
- Total volume purged
- Times of stabilization parameter measurements
- Purging rate, including any changes in rate
- Temperature measurements
- pH measurements
- Specific conductance measurements
- Dissolved oxygen measurements
- Turbidity measurements
- Site or monitoring well conditions impacting observed dissolved oxygen and turbidity measurements
- Color of groundwater
- Odor of groundwater

2.2. Record the following for Water Level and Purge Volume Determination (FS 2211):

- Depth to groundwater
- Total depth of well

- Length of water column
- Well diameter
- Volume of water in the well
- Volume of pump
- Tubing diameter
- Length of tubing
- Volume of flow cell
- Volume in the pumping system

2.3. Record the following for Well Purging (FS 2212)

- Calculations for pumping rates, including any changes in rates
- Flow meter readings
- Volume of water purged
- Placement depth of tubing or pump intake
- Depth and length of screened interval
- Time needed to purge one (1) well volume or purging equipment volume
- Well volumes or purging equipment volumes purged
- Temperature measurements
- pH measurements
- Specific conductance measurements
- Dissolved oxygen measurements
- Turbidity measurements
- Purging rate, including any changes in rate
- Drawdown in the well

3. IN-PLACE PLUMBING SOURCES INCLUDING DRINKING WATER SYSTEMS

3.1. Record the following for all samples:

- Plumbing and tap material construction (if known)
- Flow rate at which well was purged
- Amount of time well was allowed to purge
- Flow rate at time of sample collection
- Public water system identification number (if applicable)
- Name and address of water supply system and an emergency phone number for notification of sample results (if applicable)

4. SURFACE WATER SAMPLING

- Sample collection depth

- Beginning and ending times (24 hr) for timed composite sampling
- Type of composite (e.g., flow-proportioned, continuous, etc.)

5. WASTEWATER SAMPLING
   - Beginning and ending times (24 hr) for timed composite sampling
   - Type of composite (e.g. flow-proportioned, continuous, etc.)

FD 5120. RECORDS FOR NON-AQUEOUS ENVIRONMENTAL SAMPLES

Document the following information for all samples when using the indicated procedures.

FD 5130. DOCUMENTATION SPECIFIC TO SOIL SAMPLING (FS 3000)

1. GENERAL SOIL SAMPLING
   - Sample collection depth
   - Areal location of sample
   - Sample collection device
2. Sampling for Volatile Organic Compounds (VOC) per EPA Method 5035
   - Tare weight of VOC sample vial (if applicable)
   - Weight of sample (if applicable)

FD 5140. DOCUMENTATION SPECIFIC TO SEDIMENT SAMPLING (FS 4000)

1. General Sediment Sampling
   - Sample collection depth
   - Areal location of sample
   - Sample collection device
2. Sampling for Volatile Organic Compounds (VOC) per EPA Method 5035
   - Tare weight of VOC sample vial (if applicable)
   - Weight of sample (if applicable)

FD 5200. Documentation Specific to Waste Sampling (FS 5000)

1. DRUM SAMPLING

   1.1. Record the following information for each drum:

   - Type of drum and description of contents
   - Drum number, if applicable
   - Terrain and drainage condition
   - Shape, size and dimensions of drum
   - Label wording or other markings

- Dimensional extent of leaks or spills associated with the drum
- Drum location (or location map)

1.2. Record the following information for the drum sample(s):

- Description of phases, colors, crystals, powders, sludges, etc.
- Stratified layers sampled, including aliquot amounts for composites, if applicable

1.3. Record the following for field testing results on opened drums and drum samples:

- Background readings for OVA meters
- Sample readings for OVA meters
- Type of OVA probe
- Radiation background reading and sample radiation reading
- Type of radiation monitor used
- Oxygen and LEL readings from container opening
- Water reactivity results
- Specific gravity
- PCB test results
- Water solubility results
- pH of aqueous wastes
- Results of chemical test strips
- Ignitability results
- Results of other chemical hazard test kits
- Miscellaneous comments for any tests

2. Documentation for Tanks

2.1. Record the following information for the tank:

- Type of tank, tank design and material of construction of tank
- Description of tank contents and markings
- Tank number or other designation, if applicable
- Terrain and drainage condition
- Shape, size and dimensions of tank
- Label or placard wording or other markings
- Dimensional extent of leaks or spills associated with the tank
- Tank location (or location map)

2.2. Record the following information for the tank sample(s):

- Description of phases, colors, crystals, powders, sludges, etc.

- Stratified layers sampled, including aliquot amounts for composites, if applicable

2.3. Record the following for field testing results on opened tanks and tank samples:

- Background readings for OVA meters
- Sample readings for OVA meters
- Type of OVA probe
- Radiation background reading and sample radiation reading
- Type of radiation monitor used
- Oxygen and LEL level from container opening
- Water reactivity results
- Specific gravity
- PCB test results
- Water solubility results
- pH of aqueous wastes
- Results of chemical test strips
- Ignitability results
- Results of other chemical hazard test kits
- Miscellaneous comments for any tests

3. DOCUMENTATION FOR WASTE LEACHATE AND WASTE SUMP SAMPLES

3.1. Document information specific to leachate and sump sampling according to the documentation requirements for the respective DEP SOPs employed to collect samples (FS 2100, FS 2200, FS 4000, FS 5100 and FS 5200).

4. DOCUMENTATION FOR WASTE PILE SAMPLES

4.1. Document information specific to waste pile sampling according to associated regulatory requirements for the project.

5. DOCUMENTATION FOR WASTE IMPOUNDMENT AND WASTE LAGOON SAMPLES

5.1. Document information specific to impoundment and lagoon sampling according to the documentation requirements for the respective DEP SOPs employed to collect samples (FS 2100, FS 4000, FS 5100, and FS 5200).

FD 5300. Documentation for Biological Sampling

The following SOP sections list required documentation items for specific biological sampling procedures, as indicated.

FD 5310. DOCUMENTATION FOR BIOLOGICAL AQUATIC HABITAT CHARACTERIZATION

Minimum documentation required for biological habitat characterization and sampling is listed below according to requirements as specified in the indicated sampling and field-testing DEP SOPs.

FD 5311. *Physical/Chemical Characterization for Biological Sampling (FT 3001)*

1. Record the following information or use the Physical/Chemical Characterization Field Sheet (Form FD 9000-3):

- Submitting agency code
- Submitting agency name
- STORET station number
- Sample date
- Sample location including county
- Field identification
- Receiving body of water
- Time of sampling
- Percentage of land-use types in the watershed that drain to the site
- Potential for erosion within the portion of the watershed that affects the site
- Local non-point-source pollution potential and obvious sources
- Typical width of 100-meter section of river or stream
- Size of the system or the size of the sample area within the system (lake, wetland, or estuary)
- Three measurements of water depth across the typical width transect
- Three measurements of water velocity, one at each of the locations where water depth was measured
- Vegetated riparian buffer zone width on each side of the stream or river or at the least buffered point of the lake, wetland or estuary
- Presence of artificial channelization in the vicinity of the sampling location (stream or river)
- Description of state of recovery from artificial channelization
- Presence or absence of impoundments in the area of the sampling location
- Vertical distance from the current water level to the peak overflow level
- Distance of the high water mark above the stream bed
- Observed water depth at high water mark location
- Percentage range that best describes the degree of shading in the sampling area
- Any odors associated with the bottom sediments
- Presence or absence of oils in the sediment
- Any deposits in the area, including the degree of smothering by sand or silt
- Depth of each water quality measurement
- Temperature

- pH
- Dissolved oxygen
- Specific conductance
- Salinity
- Secchi depth
- Type of aquatic system sampled
- Stream magnitude (order designation)
- Description of any noticeable water odors
- Term that best describes the relative coverage of any oil on the water surface
- Term that best describes the amount of turbidity in the water
- Term that best describes the color of the water
- Weather conditions during the time of sampling
- Any other conditions/observations that are helpful in characterizing the site
- Relative abundances of periphyton, fish, aquatic macrophytes and iron/sulfur bacteria
- List and map of dominant vegetation observed
- Sampling team designation
- Signature(s) of sampler(s)
- Signature date

2. For streams and rivers, draw a grid sketch of the site (optionally use Form FD 9000-4), showing the location and amount of each substrate type (as observed by sight or touch). Using the grid sketch, count the number of grid spaces for each substrate type. Divide each of these numbers by the total number of grid spaces contained within the site sketch. Record this percent coverage value for each substrate type. If the substrates are sampled, record the number of times each substrate is sampled by an indicated method.

3. For lakes, divide the site map into twelve sections and note visual markers that will assist in distinguishing those sections.

4. Photographs of the sampling area are also useful tools for documenting habitat conditions and identifying station location.

FD 5312. *Stream and River Biological Habitat Assessment Records (FT 3100)*

1. Record the following information or use Form FD 9000-5, Stream/River Habitat Assessment Field Sheet:

- Submitting organization name and/or code
- STORET station number
- Assessment date
- Sampling location including county
- Field identification

- Receiving body of water
- Time of sampling upon arrival at the site

2. Additionally record the following:
   - Substrate diversity score
   - Substrate availability score
   - Water velocity score
   - Habitat smothering score
   - Artificial channelization score
   - Bank stability score for each bank
   - Riparian buffer zone width score for each bank
   - Riparian zone vegetation quality score for each bank
   - Primary habitat components score
   - Secondary habitat components score
   - Habitat assessment total score
   - Additional comments and observations
   - Signatures

3. Record the following information or use Form FD 9000-4, Stream/River Habitat Sketch Sheet for each 100-meter segment assessed.
   - Link to the waterbody name, location of 100-meter segment, analyst name(s) and date of the assessment
   - Code, symbol or icon used to map each substrate observed in the segment
   - Proportionate sketch or map of the abundance of each habitat (substrate) observed in the 100-meter segment, oriented to the direction of flow
   - Location of velocity measurements taken within the segment
   - Location of habitats smothered by sand or silt
   - Location of unstable, eroding banks
   - Locations along the segment where the natural, riparian vegetation is altered or eliminated
   - Plant taxa observed
   - Additional notes and observations

FD 5313. *Lake Biological Habitat Assessment Records (FT 3200)*

1. Document the following information or use the Lake Habitat Assessment Field Sheet (Form FD 9000-6):
   - STORET station number

- Sampling date
- Sampling location including lake name
- Eco-region
- Field identification number
- County name
- Lake size
- Features observed
- Description of the hydrology of the system (water residence time)
- Lake water color
- Secchi depth score
- Vegetation quality score
- Stormwater inputs score
- Bottom substrate quality score
- Lakeside adverse human alterations score
- Upland buffer zone score
- Adverse watershed land use score
- Habitat assessment total score
- Additional comments and observations
- Name and Signature of analyst

FD 5320. BIOLOGICAL AQUATIC COMMUNITY SAMPLING RECORDS (FS 7000)

Minimum documentation required for biological sampling for procedures described in FS 7000 is listed below according to requirements as specified in the indicated sampling DEP SOPs.

FD 5321. *Periphyton Sampling Records (FS 7200)*

For each sample, record the following:

- Station sampled
- Date collected

FD 5322. *Qualitative Periphyton Sampling Records (FS 7220)*

Complete the Physical/Chemical Characterization Field Sheet (Form FD 9000-3), Stream/River Habitat Sketch Sheet (Form FD 9000-4) or site map and Stream/River Habitat Assessment Field Sheet (Form FD 9000-5), as appropriate for the water body sampled (see FT 3000 – FT 3100). Other customized formats may be used to record the information prompted on the above forms.

FD 5323. *Rapid Periphyton Survey Records (FS 7230)*

For each 100-meter reach surveyed, record the following information or use Form FD 9000-8, Rapid Periphyton Survey Field Sheet:

- Site or waterbody name
- Survey date
- Name(s) of analyst(s)
- Transect mark number (10-meter segment within the 100-meter reach)
- Transect point (1 – 9)
- Algae sample collected
- Algal thickness rank (per FS 7230 procedure)
- Algae type
- Canopy cover (per FS 7230 procedure)
- Bottom visibility
- Water color
- Additional comments or observations

FD 5324. *Lake Vegetation Index Records (FS 7310)*

Record the following information or use Form FD 9000-7, Lake Vegetation Index Data Field Sheet:

- Waterbody name
- Assessment or sampling date
- County name
- Name of analyst(s)
- STORET station number
- Signature(s) of analyst(s)
- Lake water level
- Presence of algal mats
- Lake units sampled (12-sector procedure per FS 7310)
- Taxa observed in each selected unit
- Dominant and co-dominant taxa in each unit
- Taxa collected for further identification
- Approximate water depth for each taxon collected

FD 5325. *Rapid Bioassessment (Biorecon) Records (FS 7410)*

Record the following information or use the Biorecon Field Sheet (Form FD 9000-1).

- STORET station number
- Location, including latitude and longitude
- Watershed or basin name
- Family or genus of all organisms from all material in all four dipnet sweeps
- Total taxa tallies
- Taxa richness, Ephemeroptera taxa, Trichoptera taxa, Long-lived taxa, Clinger taxa, and Sensitive taxa
- Abundance code for each taxon
- Name(s) of analysts collecting and sorting samples
- Habitat types (substrates) sampled
- Name(s) of analyst(s) performing quality control
- Signatures
- Collection date and time

FD 5326. *Stream Condition Index (D-frame Dipnet) Sampling Records (FS 7420)*

1. Complete the Physical/Chemical Characterization Field Sheet (Form FD 9000-3), Stream/River Habitat Sketch Sheet (Form FD 9000-4) or site map and Stream/River Habitat Assessment Field Sheet (Form FD 9000-5) forms appropriate for the water body sampled (see FT 3000 – FT 3400). Other customized formats may be used to record the information prompted on the above forms.
2. Record the following for each sample:
    - Number of sweeps for each habitat
    - Number of containers per sample

FD 5327. *Sediment Core Biological Grab Sampling Records (FS 7440)*

Record the sampling location of site grab core samples.

FD 5328. *Sediment Dredge Biological Grab Sampling Records (FS 7450)*

Record the sampling location of site grab dredge samples.

FD 5329. *Lake Condition Index (Lake Composite) Sediment Dredge Biological Grab Sampling Records (FS 7460)*

Record the following or use DEP Form FD 9000-2 (Composite Lake Sampling Sheet):

- Sampling date
- Lake name
- Sampling equipment used
- Comments and observations

- Dredge drop number (1 – 12)
- Sampling depth for each drop number
- Sampling location of site grab dredge sample for each drop (include lake sector map)
- Sediment type(s) in grab dredge sample for each drop
- Location of any water quality measurements

FD 6000. QUALITY CONTROL DOCUMENTATION

1. Document all field quality control samples in the permanent field records.
2. At a minimum, record the following information:
   - The type, time and date that the quality control sample was collected; and
   - The preservative(s) (premeasured or added amount) and preservation checks performed.
3. If blanks are collected/prepared by the field organization, maintain records of the following:
   - Type of analyte-free water used;
   - Source of analyte-free water (include lot number if commercially purchased);
   - A list of the sampling equipment used to prepare the blank.

   If items above are specified in an internal SOP, you may reference the SOP number and revision date in the field notes. Note any deviations to the procedure in the field notes.
4. For trip blanks, record the following:
   - Date and time of preparation
   - Storage conditions prior to release to the sample collecting organization
   - Type of analyte-free water used
   - Source and lot number (if applicable) of analyte-free water

   4.1. Include trip blank information in the sampling kit documentation per FD 2000, section 2.
5. For duplicates, record the technique that was used to collect the sample.
6. For split samples, identify the method used to collect the samples and the source(s) of the sample containers and preservatives.

FD 7000. LEGAL OR EVIDENTIARY DOCUMENTATION

1. Scope: The use of legal or evidentiary Chain-of-Custody (COC) protocols is not usually required by DEP, except for cases involving civil or criminal enforcement. Do not use these procedures for routine sampling for compliance, for example, unless evidentiary custody protocols are specifically mandated in a permit or other legal order or when required for enforcement actions.
2. <u>General Procedural Instructions</u>

   2.1. Follow applicable requirements in FD 1000 – FD 5000 for all evidence samples.

2.2. Establish and maintain the evidentiary integrity of samples and/or sample containers. Demonstrate that the samples and/or sample containers were handled and transferred in such a manner as to eliminate possible tampering.

2.2.1. Document and track all time periods and the physical possession and storage of sample containers and samples from point of origin through the final analytical result and sample disposal.

FD 7100. General Requirements for Evidentiary Documentation

1. CHAIN OF CUSTODY RECORDS: Use the Chain-of-Custody (COC) records to establish an intact, contiguous record of the physical possession, storage, and disposal of sample containers, collected samples, sample aliquots, and sample extracts or digestates. For ease of discussion, the above-mentioned items are referred to as “samples”.

1.1. Account for all time periods associated with the physical samples.

1.2. Include signatures of all individuals who physically handle the samples.

1.2.1. The signature of any individual on any record that is designated as part of the Chain-of-Custody is their assertion that they personally handled or processed the samples identified on the record.

1.2.2. Denote each signature with a short statement that describes the activity of the signatory (e.g., “sampled by”, “received by”, “relinquished by”, etc.).

1.2.3. In order to simplify recordkeeping, minimize the number of people who physically handle the samples.

2. CONSOLIDATION OF RECORDS: The COC records need not be limited to a single form or document. However, limit the number of documents required to establish COC, where practical, by grouping information for related activities in a single record. For example, a sample transmittal form may contain both certain field information and the necessary transfer information and signatures for establishing delivery and receipt at the laboratory.

3. LIABILITY FOR CUSTODY DOCUMENTATION: Ensure appropriate personnel initiate and maintain sample chain-of-custody at specified times.

3.1. Begin legal chain-of-custody when the precleaned sample containers are dispatched to the field.

3.1.1. Omit the transmittal record for precleaned sample containers if the same party provides the containers and collects the samples.

3.2. Sign the COC record upon relinquishing the prepared sample kits or containers.

3.3. Sign the COC record upon receipt of the sample kits or containers.

3.4. Thereafter, ensure that all parties handling the samples maintain sample custody (i.e., relinquishing and receiving) and documentation until the samples or sampling kits are relinquished to a common carrier.

3.4.1. The common carrier should not sign COC forms.

3.4.2. Indicate the name of the common carrier in the COC record, when used. Retain shipping bills and related documents as part of the record.

3.4.3. Ensure that all other transferors and transferees releasing or accepting materials from the common carrier sign the custody record.

3.5. Chain-of-custody is relinquished by the party who seals the shipping container and is accepted by the party who opens it.

3.5.1. Indicate the date and time of sealing of the transport container for shipment.

3.5.2. See FD 7200, section 3 below regarding the use of custody seals.

4. SAMPLE SHIPPING OR TRANSPORTING

4.1. Affix tamper-indicating custody seals or evidence tape before shipping samples.

4.1.1. Seal sample container caps with tamper-indicating custody seals or evidence tape before packing for shipping or transport.

4.1.2. Seal sample transport or shipping containers with strapping tape and tamper-indicating custody seals or evidence tape.

4.1.3. If the same party collects then possesses (or securely stores), packs and transports the samples from time of collection, omit any use of custody seals or evidence tape.

4.2. Keep the COC forms with the samples during transport or shipment. Place the COC records in a waterproof closure inside the sealed ice chest or shipping container.

FD 7200. Required Documentation for Evidentiary Custody

1. GENERAL CONTENT REQUIREMENTS: Document the following in COC tracking records by direct entry or linkage to other records:

- Time of day and calendar date of each transfer or handling procedure
- Signatures of transferors, transferees and other personnel handling samples
- Location of samples (if stored in a secured area)
- Description of all handling procedures performed on the samples for each time and date entry recorded above
- Storage conditions for the samples, including chemical preservation and refrigeration or other cooling
- Unique identification for all samples
- Final disposition of the physical samples
- Common carrier identity and related shipping documents

2. DOCUMENTATION CONTENT FOR SAMPLE TRANSMITTAL

Provide a Chain-of-Custody record for all evidentiary samples and subsamples that are transmitted or received by any party. Include the following information in the COC record of transmittal:

- Sampling site name and address
- Date and time of sample collection
- Unique field identification code for each sample source and each sample container
- Names of personnel collecting samples
- Signatures of all transferors and transferees

- Time of day and calendar date of all custody transfers
- Clear indication of number of sample containers
- Required analyses by approved method number or other description
- Common carrier usage
- Sample container/preservation kit documentation, if applicable

3. CHAIN-OF-CUSTODY SEALS: If required, affix tamper-indicating evidence tape or seals to all sample, storage and shipping container closures when transferring or shipping sample container kits or samples to another party.

    3.1. Place the seal so that the closure cannot be opened without breaking the seal.

    3.2. Record the time, calendar date, and signatures of responsible personnel affixing and breaking all seals for each sample container and shipping container.

    3.3. Affix new seals every time a seal is broken until continuation of evidentiary custody is no longer required.

FD 7300. Documenting Controlled Access to Evidence Samples

Control and document access to all evidentiary samples and subsamples with adequate tracking. Documentation must include records about each of the activities and situations listed below, when applicable to sample evidence, and must track the location and physical handling of all samples by all persons at all times. See FS 1000 for additional discussion about procedures for handling evidence samples.

1. Limit the number of individuals who physically handle the samples as much as practicable.

2. When storing samples and subsamples, place samples in locked storage (e.g., locked vehicle, locked storeroom, etc.) at all times when not in the possession or view of authorized personnel.

3. Alternatively, maintain restricted access to facilities where samples are stored. Ensure that unauthorized personnel are not able to gain access to the samples at any time.

4. Do not leave samples in unoccupied motel or hotel rooms or other areas where access cannot be controlled by the person(s) responsible for custody without first securing samples and shipping or storage containers with tamper-indicating evidence tape or custody seals.

FD 7400. Documenting Disposal of Evidence Samples

1. Dispose of the physical samples only with the concurrence of the affected legal authority, sample data user, and/or submitter/owner of the samples.

2. Record all conditions of disposal and retain correspondence between all parties concerning the final disposition of the physical samples.

3. Record the date of disposal, the nature of disposal (i.e., sample depleted, sample flushed into sewer, sample returned to client, etc.), and the name of the individual who performed the disposal. If samples are transferred to another party, document custody transfer in the same manner as other transfers (see FD 7000 – FD 7200).

FD 8000. (RESERVED)

FD 9000. FORMS

Forms to facilitate documentation of sampling, field-testing, and biological laboratory calculation activities are available on the Department's website. These forms are for unrestricted public use and are presented in example formats. *The use of these forms is not mandatory. However,* ***some*** *of the data elements and other information denoted by the form prompts comprise* ***required documentation*** *items*. *Not all required documentation is illustrated in the form examples.* Customize these forms as needed. These forms are available as separate document files. The following forms are incorporated into the indicated SOPs for convenience of use:

- Form FD 9000-1 Biorecon Field Sheet (FS 7000)
- Form FD 9000-2 Composite Lake Sampling Sheet for <1000 Acres (FS 7000)
- Form FD 9000-3 Physical/Chemical Characterization Field Sheet (FT 3000)
- Form FD 9000-4 Stream/River Habitat Sketch Sheet (FT 3000)
- Form FD 9000-5 Stream/River Habitat Assessment Field Sheet (FT 3000)
- Form FD 9000-6 Lake Habitat Assessment Field Sheet (FT 3000)
- Form FD 9000-7 Lake Vegetation Index Data Field Sheet (FS 7000)
- Form FD 9000-8 Rapid Periphyton Survey Field Sheet (FS 7000)

FQ 1000. FIELD QUALITY CONTROL REQUIREMENTS

Field quality control measures monitor the sampling event to ensure that the collected samples are representative of the sample source.

Field-collected blanks must demonstrate that the collected samples have not been contaminated by:

- The sampling environment
- The sampling equipment
- The sample container
- The sampling preservatives
- Sample transport
- Sample storage

FQ 1100. Sample Containers

Sample containers must be free from contamination by the analytes of interest or any interfering constituents and must be compatible with the sample type.

FQ 1200. Sampling Operations

1. When collected, analyze all quality control samples for the same parameters as the associated samples.

   1.1. When collected, collect blanks for the following parameter groups and tests:

   - Volatile Organics
   - Extractable Organics
   - Metals
   - Ultratrace Metals
   - Inorganic Nonmetallics
   - Radionuclides
   - Petroleum Hydrocarbons and Oil & Grease
   - Volatile Inorganics
   - Aggregate Organics except Biochemical Oxygen Demand

   1.2. Blanks are not required for:

   - Microbiological (all types)
   - Toxicity
   - Field parameters such as pH, Specific Conductance, Residual Chlorine, Temperature, Light Penetration, Dissolved Oxygen, ORP and Salinity
   - Radon

- Algal Growth Potential
- Biological Community
- Physical and Aggregate Properties
- Biochemical Oxygen Demand

2. Preserve, transport, document and handle all quality control samples as if they were samples. Once collected, they must remain with the sample set until the laboratory has received them.

3. Except for trip blanks, prepare all quality control samples **on-site in the field**.

   3.1. Do not prepare precleaned equipment blanks in advance at the base of operations.

   3.2. Do not prepare field-cleaned equipment blanks after leaving the sampling site.

4. Perform and document any field QC measures specified by the analytical method (such as trip blanks for volatile organics).

FQ 1210. QUALITY CONTROL BLANKS

FQ 1211. *Precleaned Equipment Blanks*

1. USE: Monitors on-site sampling environment, sampling equipment decontamination, sample container cleaning, the suitability of sample preservatives and analyte-free water, and sample transport and storage conditions for water, waste, soil, or sediment samples.

2. Collect these blanks using sampling equipment that has been brought to the site precleaned and ready for use. The cleaning procedures used for the blank collection must be identical to those used for the field sample collection.

3. Collect these blanks before the equipment set has been used.

4. Prepare equipment blanks by rinsing the sampling equipment set with the appropriate type of analyte-free water and collecting the rinse water in appropriate sample containers (see FQ 1100).

FQ 1212. *Field-Cleaned Equipment Blanks*

1. USE: Monitors on-site sampling environment, sampling equipment decontamination, sample container cleaning, the suitability of sample preservatives and analyte-free water, and sample transport and storage conditions.

2. Collect these blanks using sampling equipment that has been cleaned in the field (i.e., between sampling points). The cleaning procedures used for the blank collection must be identical to those used for the field sample collection.

3. Prepare field-cleaned equipment blanks immediately after the equipment is cleaned in the field and before leaving the sampling site.

4. Prepare equipment blanks by rinsing the sampling equipment set with the appropriate type of analyte-free water and collecting the rinse water in appropriate sample containers (see FQ 1100).

   4.1. For intermediate sampling devices or equipment, site-water rinsing is defined as the decontamination step, if this is the only cleaning that will be performed on the equipment prior to collecting the sample.

4.1.1. In this case, collect the equipment blank after rinsing the intermediate device 3 times with site water

4.1.2. Follow the site-water rinses with 3 rinses using analyte-free water.

4.1.3. Collect the equipment blank with a subsequent rinse of the device using additional analyte-free water to collect sufficient blank volume.

FQ 1213. *Trip Blanks*

1. USE: Monitors sample container cleaning, the suitability of sample preservatives and analyte-free water, and sample transport and storage conditions.

2. The organization that is providing the VOC vials must provide the trip blanks by filling two or more VOC vials with analyte-free water and preservatives (if needed).

   2.1. To prevent degradation of the trip blank, long-term storage of prepared trip blanks is not recommended.

3. These blanks are applicable if samples are to be analyzed for volatile constituents (volatile organics, methyl mercury, etc.) in water, waste, soils, or sediments.

4. Place a set of trip blanks in each transport container used to ship/store empty VOC vials. They must remain with the VOC vials during the sampling episode and must be transported to the analyzing laboratory in the same shipping or transport container(s) as the VOC samples.

5. Trip blanks must be opened **only** by the laboratory after the blank and associated samples have been received for analysis.

FQ 1214. *Field Blanks*

1. USE: Monitors on-site sampling environment, sample container cleaning, the suitability of sample preservatives and analyte-free water, and sample transport and storage conditions for water, waste, soil or sediment samples.

2. Prepare field blanks by pouring analyte-free water into sample containers for each parameter set to be collected.

3. Field blanks are not required if equipment blanks (FQ 1211 or FQ 1212) are collected.

FQ 1220. FIELD DUPLICATES

1. USE: Designed to measure the variability in the sampling process.

2. GENERAL CONSIDERATIONS:

   2.1. Collect duplicates by **repeating** (simultaneously or in rapid succession) the entire sample acquisition technique that was used to obtain the first sample.

      2.1.1. Collect, preserve, transport and document duplicates in the same manner as the samples. **<u>These samples are not considered laboratory duplicates</u>**.

   2.2. When collected, analyze field duplicates for the same parameters as the associated samples.

   2.3. If possible, collect duplicate samples from sampling locations where contamination is present.

2.4. Field duplicates must be collected if required by the analytical method and as required by a DEP program.

FQ 1221. *Water Duplicates*

Collect water duplicates by sampling from successively collected volumes (i.e., samples from the next volume of sample water).

FQ 1222. *Soil Duplicates*

Collect soil duplicates from the same sample source (i.e., soil from the same soil sampling device).

FQ 1230. MANDATORY FIELD QUALITY CONTROLS

1. The respondent, permittee or contractor and the sampling organization are responsible for ensuring that blanks (excluding trip blanks) are collected at a minimum of 5% of each reported test result/matrix combination for the life of a project.

    1.1. Collect at least one blank for each reported test result/matrix combination each year for each project.

    1.2. If a party wishes to claim that a positive result is due to external contamination sources during sample collection, transport or analysis, then at least one field collected blank (excludes trip blanks) must have been collected at the same time the samples were collected and analyzed with the same sample set.

    1.3. A project will be defined by the organization responsible for collecting the samples for the project.

        1.3.1. When applicable, define the scope of the project in conjunction with the appropriate DEP authority.

2. When collecting a set of blanks, use the following criteria:

    2.1. <u>Equipment Blanks</u>:

        2.1.1. Collect field-cleaned equipment blanks if any sample equipment decontamination is performed in the field.

        2.1.2. If no decontamination is performed in the field, collect precleaned equipment blanks if the equipment is not certified clean by the vendor or the laboratory providing the equipment.

        2.1.3. Equipment blanks are not required for volatile organic compounds.

    2.2. <u>Field Blanks</u>:

        2.2.1. Collect field blanks if no equipment except the sample container is used to collect the samples or if the sampling equipment is certified clean by the vendor or the laboratory providing the equipment.

            2.2.1.1. If a sample container is used as an intermediate sample collection device, collect an equipment blank by rinsing the decontaminated collection container as the substitute for the field blank.

        2.2.2. Field blanks are not required for volatile organic compounds.

    2.3. <u>Trip Blanks:</u>

2.3.1. These blanks are applicable if samples are to be analyzed for volatile organic compounds. See FQ 1213 for frequency, preparation and handling requirements.

3. OPTIONAL QUALITY CONTROL MEASURES

3.1. The method or project may require collection of additional quality control measures as outlined in FQ 1210 (Blanks), FQ 1220 (Duplicates) and FQ 1240 (Split Samples).

FQ 1240. SPLIT SAMPLES

The DEP or the client may require split samples as a means of determining compliance or as an added measure of quality control. Unlike duplicate samples that measure the variability of both the sample collection and laboratory procedures, split samples measure only the variability **between** laboratories. Therefore, the laboratory samples must be subsamples of the same parent sample and every attempt must be made to ensure sample homogeneity.

Collect, preserve, transport and document split samples using the same protocols as the related samples. In addition, attempt to use the same preservatives (if required).

If split samples are incorporated as an added quality control measure, the DEP recommends that all involved parties agree on the logistics of collecting the samples, the supplier(s) of the preservatives and containers, the analytical method(s), and the statistics that will be used to evaluate the data.

FQ 1241. *Soils, Sediments, Chemical Wastes and Sludges*

Collecting split samples for these matrices is not recommended because a true split sample in these matrices is not possible.

FQ 1242. *Water*

Collect split samples for water in one of two ways:

1. Mix the sample in a large, appropriately precleaned, intermediate vessel (a churn splitter is recommended). This method shall not be used if volatile or extractable organics, oil and grease or total petroleum hydrocarbons are of interest. While continuing to thoroughly mix the sample, pour aliquots of the sample into the appropriate sample containers. Alternatively:

2. Fill the sample containers from consecutive sample volumes **from the same sampling device**. If the sampling device does not hold enough sample to fill the sample containers, use the following procedure:

2.1. Fill the first container with half of the sample, and pour the remaining sample into the second container.

2.2. Obtain an additional sample, pour the first half into the **second** container, and pour the remaining portion into the first container.

2.3. Continue with steps described in sections 2.1 and 2.2 above until both containers are filled.

FQ 1250. QUALITY CONTROL DOCUMENTATION

1. Document all field quality control samples in the permanent field records.

2. At a minimum, record the following information:

- The type, time and date that the quality control sample was collected; and
- The preservative(s) (premeasured or added amount) and preservation checks performed.

3. If blanks are collected/prepared by the field organization, maintain records of the following:
   - Type of analyte-free water used;
   - Source of analyte-free water (include lot number if commercially purchased);
   - A list of the sampling equipment used to prepare the blank.

   If items above are specified in an internal SOP, you may reference the SOP number and revision date in the field notes. Note any deviations to the procedure in the field notes.

4. For trip blanks, record the following:
   - Date and time of preparation
   - Storage conditions prior to release to the sample collecting organization
   - Type of analyte-free water used
   - Source and lot number (if applicable) of analyte-free water

   4.1. Include trip blank information in the sampling kit documentation per FD 2000, section 2.

5. For duplicates, record the technique that was used to collect the sample.

6. For split samples, identify the method used to collect the samples and the source(s) of the sample containers and preservatives.

FS 1000. GENERAL SAMPLING PROCEDURES

See also the following Standard Operating Procedures:

- FA 1000 and 2000 Administrative Procedures
- FC 1000 Cleaning/Decontamination Procedures
- FD 1000-9000 Documentation Procedures
- FM 1000 Field Planning and Mobilization
- FQ 1000 Field Quality Control Requirements

FS 1001. *Preliminary Activities*

1. Begin each sampling trip with some planning and coordination. Refer to FM 1000 for recommendations and suggestions on laboratory selection and communication, and field mobilization.

   1.1. DEP recommends that a minimum of two people be assigned to a field team. In addition to safety concerns, the process of collecting the samples, labeling the containers and completing the field records is much easier if more than one person is present.

   1.2. If responding to incidents involving hazardous substances, DEP recommends that four or five people be assigned to the team.

2. EQUIPMENT

   2.1. Select appropriate equipment based on the sampling source (see FS 2000 to FS 8200), the analytes of interest and the sampling procedure.

      2.1.1. If properly cleaned, sample containers may be used as collection devices or intermediate containers.

   2.2. The equipment construction must be consistent with the analytes or analyte groups to be collected (see Tables FS 1000-1 and FS 1000-2).

   2.3. Bring precleaned equipment to the field or use equipment that has been certified clean by the vendor or laboratory.

3. DEDICATED EQUIPMENT STORAGE

   3.1. Store all dedicated equipment (except dedicated pump systems or dedicated drop pipes) in a controlled environment.

   3.2. If possible, store equipment in an area that is located away from the sampling site. If equipment other than dedicated pumps or dedicated drop pipes is stored in monitoring wells, suspend the equipment above the formation water.

   3.3. Securely seal the monitoring well in order to prevent tampering between sampling events.

   3.4. Decontaminate all equipment (except dedicated pumps or drop pipes) before use according to the applicable procedures in FC 1000.

4. SAMPLE CONTAINERS

   4.1. The analyses to be performed on the sample determine the construction of sample containers.

4.2. Inspect all containers and lids for flaws (cracks, chips, etc.) before use. Do not use any container with visible defects or discoloration.

FS 1002. *Contamination Prevention and Sample Collection Order*

1. CONTAMINATION PREVENTION

1.1. Take special effort to prevent cross contamination and contamination of the environment when collecting samples. Protect equipment, sample containers and supplies from accidental contamination.

1.1.1. Do not insert pump tubing, measurement probes, other implements, fingers, etc. into sample containers or into samples that have been collected for laboratory analysis.

1.1.1.1. If it is necessary to insert an item into the container or sample, ensure that the item is adequately decontaminated for the analytes of interest to be analyzed in the sample.

1.1.2. If possible, collect samples from the least contaminated sampling location (or background sampling location) to the most contaminated sampling location.

1.1.2.1. Collect the ambient or background samples first and store them in separate ice chests or shipping containers.

1.1.3. Collect samples in flowing water from downstream to upstream.

1.1.4. Do not store or ship highly contaminated samples (concentrated wastes, free product, etc.) or samples suspected of containing high concentrations of contaminants in the same ice chest or shipping container with other environmental samples.

1.1.4.1. Isolate these sample containers by sealing them in separate, untreated plastic bags immediately after collecting, preserving, labeling, etc.

1.1.4.2. Use a clean, untreated plastic bag to line the ice chest or shipping container.

2. SAMPLE COLLECTION ORDER

2.1. Sampling order is a recommendation to be modified depending on site circumstances. Unless field conditions justify other sampling regimens, collect samples in the following order:

- Volatile Organics and Volatile Inorganics
- Extractable Organics, Petroleum Hydrocarbons, Aggregate Organics and Oil & Grease
- Total Metals
- Dissolved Metals
- Inorganic Nonmetallics, Physical and Aggregate Properties, and Biologicals
- Radionuclides
- Microbiological

Note: If the pump used to collect groundwater samples cannot be used to collect volatile or extractable organics, then collect all other parameters, withdraw the pump and tubing, and collect the volatile and extractable organics.

3. COMPOSITE SAMPLES

3.1. Do not collect composite samples unless required by permit or DEP program.

3.2. If compositing is required, use the following procedure:

3.2.1. Select sampling points from which to collect each aliquot.

3.2.2. Using the appropriate sampling technique, collect equal aliquots (same sample size) from each location and place in a properly cleaned container.

3.2.3. Record the approximate amount of each aliquot (volume or weight).

3.2.4. Add preservative(s), if required.

3.2.5. Label container and make appropriate field notes (see FD 1000-9000).

3.2.6. Notify the laboratory that the sample is a composite sample.

3.2.7. When collecting soil or sediment samples, combine the aliquots of the sample directly in the sample container with no pre-mixing. Notify the laboratory that the sample is an unmixed composite sample, and request that the laboratory thoroughly mix the sample before sample preparation or analysis.

3.2.8. When collecting water composites see FS 2000, section 1.3 or pertinent sections of other water matrix SOPs for specific details on collection.

FS 1003. *Protective Gloves*

1. Gloves serve a dual purpose to:

- Protect the sample collector from potential exposure to sample constituents
- Minimize accidental contamination of samples by the collector

2. The DEP recommends wearing protective gloves when conducting all sampling activities. They must be worn except when:

- The sample source is considered to be non-hazardous
- The samples will not be analyzed for trace constituents
- The part of the sampling equipment that is handled without gloves does not contact the sample source

3. Do not let gloves come into contact with the sample or with the interior or lip of the sample container.

4. Use clean, new, unpowdered and disposable gloves.

4.1. DEP recommends latex gloves, however, other types of gloves may be used as long as the construction materials do not contaminate the sample or if internal safety protocols require greater protection.

4.2. Note that certain materials (as might be potentially present in concentrated effluent) may pass through certain glove types and be absorbed in the skin. Many vendor catalogs provide information about the permeability of different gloves and the circumstances under which the glove material might be applicable.

4.3. The powder in powdered gloves can contribute significant contamination and DEP does not recommend wearing powdered gloves unless it can be demonstrated that the powder does not interfere with the sample analysis.

5. If gloves are used, change:
    - After preliminary activities such as pump placement;
    - After collecting all the samples at a single sampling point; or
    - If torn, or used to handle extremely dirty or highly contaminated surfaces.
6. Properly dispose of all used gloves.

FS 1004. *Container and Equipment Rinsing*

When collecting aqueous samples, rinse the sample collection equipment with a portion of the sample water before taking the actual sample. Sample containers do not need to be rinsed. In the case of petroleum hydrocarbons, oil & grease or containers with premeasured preservatives, the sample containers cannot be rinsed.

FS 1005. *Fuel-Powered Equipment and Related Activities*

1. Place all fuel-powered equipment away from, and downwind of, any site activities (e.g., purging, sampling, decontamination). If field conditions preclude such placement (i.e., the wind is from the upstream direction in a boat), place the fuel source(s) as far away as possible from the sampling activities and describe the conditions in the field notes.

2. Handle fuel (i.e., filling vehicles and equipment) prior to the sampling day. If such activities must be performed during sampling, the personnel must wear disposable gloves. Dispense all fuels, dispose of gloves downwind, and well away from the sampling activities.

3. If sampling at active gas stations, stop sample collection activities during fuel deliveries.

FS 1006. *Preservation, Holding Times and Container Types*

1. Preserve all samples according to the requirements specified in Tables FS 1000-4 through FS 1000-10.

    1.1. The information listed in the above-referenced tables supersedes any preservation techniques, holding time or container type that might be discussed in individual analytical methods.

    1.2. If samples are collected only for total phosphorus and are not for NPDES compliance, thermal preservation (ice) is not required if the sample containers are pre-preserved with acid.

2. The preservation procedures in the referenced tables specify immediate preservation. "Immediate" is defined as "within 15 minutes of sample collection." Perform all preservation on-site (in the field).

    2.1. Preservation is not required if samples can be transported back to the laboratory within 15 minutes of collecting the sample and

        2.1.1. The laboratory begins sample analysis within the 15-minute window and documents the exact time the analysis began, or

        2.1.2. The laboratory adds the appropriate preservatives (including thermal preservation) within 15 minutes of sample collection and documents the exact time that the preservation was done.

3. PRESERVING COMPOSITE WATER SAMPLES

3.1. If the sample preservation requires thermal preservation (e.g., <6°C), the samples must be cooled to the specified temperature.

3.1.1. Manually collected samples to be composited must be refrigerated at a temperature equal to or less than the required temperature.

3.1.2. Automatic samplers must be able to maintain the required temperature by packed ice or refrigeration.

3.2. When chemical preservation is also required, begin the preservation process within 15 minutes of the last collected sample.

3.3. Holding Times for Automatic Samplers:

3.3.1. If the collection period is 24 hours or less, the holding time begins at the last scheduled sample collection;

3.3.2. If the collection period exceeds 24 hours, the holding time begins with the time that the first sample is collected.

4. PH ADJUSTED PRESERVATION - Check the pH of pH-adjusted samples according to these frequencies:

4.1. During the first sampling event at a particular site, check **all** samples (includes each groundwater monitoring well, surface water location, or influent/effluent sampling location) that are pH-adjusted except volatile organics.

4.2. During subsequent visits to a particular site, check at least one sample per parameter group that must be pH-adjusted.

4.3. If the frequency of sample collection at a specified location is greater than once per month (i.e., weekly or daily), check the pH of at least one sample per parameter group (except volatile organics) according to the following schedule:

4.3.1. Weekly sampling: 1 pH check per month

4.3.2. Daily sampling: 1 pH check per week

4.4. If the frequency of sample collection at a specified location is once per month, check the pH of at least one sample per parameter group (except volatile organics) quarterly.

4.5. If site conditions vary from sampling event to sampling event, perform pH checks at increased intervals.

5. THERMAL PRESERVATION

5.1. When preservation requirements indicate cooling to a specific temperature, samples must be placed in wet ice within 15 minutes of sample collection (see 1006, section 2 above). Unless specified, do not freeze samples.

5.2. All supplies (ice, dry ice, etc.) necessary to meet a thermal preservation requirement must be onsite for immediate use.

5.3. Ship samples in wet ice. If samples are cooled to the required temperature before shipment, samples may be shipped with frozen ice packs if the specified temperature is maintained during shipment. The sample temperature must not exceed the specified temperature.

5.4. If immediate freezing is required, dry ice must be available in the field to begin the freezing process.

FS 1007. *Preventive and Routine Maintenance*

Preventive maintenance activities are necessary to ensure that the equipment can be used to obtain the expected results and to avoid unusable or broken equipment while in the field. Equipment is properly maintained when:

- It functions as expected during mobilization; and
- It is not a source of sample contamination (e.g., dust).

1. Follow the manufacturer's suggested maintenance activities and document all maintenance. At a minimum, DEP recommends the activities listed on Table FS 1000-12.

2. Maintain documentation for the following information for each piece of equipment or instrumentation. See FD 3000 also.

   2.1. Designate the identity of specific instrumentation in the documentation with a unique description or code for each instrument unit employed. This identifier may include a manufacturer name, model number, serial number, inventory number or other unique identification.

   2.2. Log all maintenance and repair performed for each instrument unit, including routine cleaning procedures and solution or parts replacement for instrument probes.

   2.3. Include the calendar date for the procedures performed.

   2.4. Record names of personnel performing the maintenance or repair tasks.

   2.5. Describe any malfunctions necessitating repair or service.

   2.6. Retain vendor service records for all affected instruments.

   2.7. Record the following for rented equipment:

   - Rental date(s)
   - Equipment type and model or inventory number or other description

   2.8. Retain the manufacturer’s operating and maintenance instructions.

FS 1008. *Documentation and References*

1. REFERENCES: All sampling references must be available for consultation in the field. These include:

   - DEP SOPs;
   - Internal SOPs;
   - Sampling and analysis plans; and/or
   - Quality Assurance Project Plans.

2. DOCUMENTATION: Complete and sign all documentation (see FD 1000).

FS 1009. *Sample Documentation and Evidentiary Custody*

1. SAMPLE DOCUMENTATION

   1.1. Document all activities related to a sampling event, including sample collection, equipment calibration, equipment cleaning and sample transport.

1.2. The required documentation related to each sampling or other field activity is specified in the associated SOPs; i.e., FQ 1000, FC 1000, the FS series, and the FT series.

1.3. The documentation requirements are also summarized in FD 1000, Field Documentation. FD 1000 additionally contains a list of example forms published with the SOPs that may be used to document various activities or as templates for creating customized forms.

2. LEGAL CHAIN OF CUSTODY (COC)

The use of legal or evidentiary Chain-of-Custody (COC) protocols is not usually required by DEP, except for cases involving civil or criminal enforcement. Do not use these procedures for routine sampling for compliance unless evidentiary custody protocols are specifically mandated in a permit or other legal order or when required for enforcement actions.

Evidentiary sample custody protocols are used to demonstrate that the samples and/or sample containers were handled and transferred in such a manner as to eliminate possible tampering.

When a client or situation requires legal COC, use the procedures in FD 7000 to document and track all time periods associated with the physical possession and storage of sample containers, samples, and subsamples from point of origin through the final analytical result and sample disposal.

When legal or evidentiary COC is required, samples must be:

- In the actual possession of a person who is authorized to handle the samples (e.g., sample collector, laboratory technician);
- In the view of the same person after being in their physical possession;
- Secured by the same person to prevent tampering; or
- Stored in a designated secure area.

2.1. Control and document access to all evidentiary samples and subsamples with adequate tracking. Documentation must include records about each of the activities and situations listed below, when applicable to sample evidence, and must track the location and physical handling of all samples by all persons at all times.

2.1.1. Limit the number of individuals who physically handle the samples as much as practicable.

2.1.2. When storing samples and subsamples, place samples in locked storage (e.g., locked vehicle, locked storeroom, etc.) at all times when not in the possession or view of authorized personnel.

2.1.3. Alternatively, maintain restricted access to facilities where samples are stored. Ensure that unauthorized personnel are not able to gain access to the samples at any time.

2.1.4. Do not leave samples in unoccupied motel or hotel rooms or other areas where access cannot be controlled by the person(s) responsible for custody without first securing samples and shipping or storage containers with tamper-indicating evidence tape or custody seals. Ice chests or other storage containers used to store sample containers in hotel rooms may be sealed instead of sealing each sample container stored within.

2.2. Use a Chain of Custody form or other transmittal record to document sample transfers to other parties. Other records and forms may be used to document internal activities if they meet the requirements for legal chain of custody.

2.3. Legal COC begins when the precleaned sample containers are dispatched to the field.

2.3.1. The person who relinquishes the prepared sample kits or containers and the individual who receives the sample kits or containers must sign the COC form unless the same party provides the containers and collects the samples.

2.3.2. All parties handling the empty sample containers and samples are responsible for documenting sample custody, including relinquishing and receiving samples, except commercial common carriers.

2.4. Shipping Samples under Legal COC

2.4.1. Complete all relevant information on the COC transmittal form or record (see FD 7200, section 2).

2.4.2. Internal records must document the handling of the samples and shipping containers in preparation for shipment. The names of all persons who have prepared the shipment must be recorded. All time intervals associated with handling and preparation must be accounted for.

2.4.3. Place the forms in a sealed waterproof bag and place in the shipping container with the samples.

2.4.4. Seal the shipping container with tamper-proof seals (see 2.6 below) so that any tampering can be clearly seen by the individual who receives the samples.

2.4.5. Note: The common carrier does not sign COC records. However, the common carrier (when used) must be identified.

2.5. Delivering Samples to the Laboratory

2.5.1. All individuals who handle and relinquish the sample containers must sign the transmittal form. The legal custody responsibilities of the field operations end when the samples are relinquished to the laboratory.

2.6. Chain of Custody Seals: If required, affix tamper-indicating evidence tape or seals to all sample, storage and shipping container closures when transferring or shipping sample container kits or samples to another party.

2.6.1. Place the seal so that the closure cannot be opened without breaking the seal.

2.6.2. Record the time, calendar date and signatures of responsible personnel affixing and breaking all seals for each sample container and shipping container.

2.6.3. Affix new seals every time a seal is broken until continuation of evidentiary custody is no longer required.

FS 1010. *Health and Safety*

Implement all local, state and federal requirements relating the health and safety.

FS 1011. *Hazardous Wastes*

Follow all local, state and federal requirements pertaining to the storage and disposal of any hazardous or investigation-derived wastes.

1. Properly manage all investigation-derived waste (IDW) so contamination is not spread into previously uncontaminated areas.

   1.1. IDW includes all water, soil, drilling mud, decontamination wastes, discarded personal protective equipment (PPE), etc. from site investigations, exploratory borings, piezometer and monitoring well installation, refurbishment, and abandonment, and other investigative activities. Containerize the IDW at the time it is generated.

   1.2. Determine if the IDW must be managed as Resource Conservation and Recovery Act (RCRA) regulated hazardous waste through appropriate testing or generator knowledge. Manage all IDW that is determined to be RCRA regulated hazardous waste according to the local state and federal requirements.

   1.3. Properly dispose of IDW that is not a RCRA-regulated hazardous waste but is contaminated above the Department's Soil Cleanup Target Levels or the state standards and/or minimum criteria for ground water quality.

   1.4. IDW that is not contaminated or contains contaminants below the Department's Soil Cleanup Target Levels or the state standards and/or minimum criteria for ground water quality may be disposed of onsite as long as the IDW will not cause a surface water violation.

   1.5. Maintain all containers holding IDW in good condition:

      1.5.1. Periodically inspect the containers for damage

      1.5.2. Ensure that all required labeling (DOT, RCRA, etc.) are clearly visible.

Appendix FS 1000
Tables, Figures and Forms

Table FS 1000-1

Equipment Construction Materials

Construction Material[1]	Acceptable Analyte Groups	Precautions
Metals		
316 Stainless Steel	All analyte groups. Recommended for inorganic nonmetallics, metals, volatile and extractable organics.	Do not use if weathered, corroded or pitted.[2]
300-Series Stainless Steel (304, 303, 302)	Suitable for all analyte groups (if used, check for corrosion before use). Recommended for inorganic nonmetallics, metals, volatile and extractable organics.	Do not use if weathered, corroded or pitted.[2] If corroded, there is a potential for samples to be contaminated with iron, chromium, copper or nickel. Check for compatibility with water chemistry for dedicated applications. Do not use in low pH, high chloride, or high TDS waters.
Low Carbon Steel Galvanized Steel Carbon Steel	Inorganic nonmetallics only.	Coring devices are acceptable for all analyte groups **if** appropriate liners are used. Use Teflon liners for organics. Use plastic or Teflon liners for metals. Do not use if weathered, corroded or pitted.[2] If corroded, there is a potential for samples to be contaminated with iron and manganese. Galvanized equipment will also contaminate with zinc and cadmium. If used to collect large samples (e.g., dredges), collect organic and metal samples may be collected from portions of the interior of the collected material.
Brass	Inorganic nonmetallics only.	Do not use if weathered, corroded or pitted.[2]
Plastics[3]		
Teflon and other fluorocarbon polymers	All analyte groups. Especially recommended for trace metals and organics.	Easily scratched. Do not use if scratched or discolored.
Polypropylene Polyethylene (All Types)	All analyte groups.	Easily scratched. Do not use if scratched or discolored.
Polyvinyl chloride (PVC)	All analyte groups except extractable and volatile organics.	Do not use when collecting extractable or volatile organics samples.

Table FS 1000-1

Equipment Construction Materials

Construction Material[1]	Acceptable Analyte Groups	Precautions
Tygon, Silicone, Neoprene	All analyte groups except extractable and volatile organics.	Do not use when collecting extractable or volatile organic samples. Do not use silicone if sampling for silica.
Viton	All analyte groups except extractable and volatile organics.[4]	Minimize contact with sample. Use only if no alternative material exists.
Glass		
Glass, borosilicate	All analyte groups except silica and boron.	

Adapted from USGS Field Manual, Chapter 2, January 2000.

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[1] Refers to construction material of the portions of the sampling equipment that come in contact with the sample (e.g., housing of variable speed submersible pump must be stainless steel if extractable organics are sampled; the housing of a variable speed submersible pump used to sample metals may be plastic.)

[2] Corroded/weathered surfaces are active sorption sites for organic compounds.

[3] Plastics used in connection with inorganic trace element samples (including metals) must be uncolored or white.

[4] May be allowable for specialized parts where no alternative material exists (e.g., Viton seals are the best available seal for some dedicated pump systems), however, contact with the sample must be minimized.

Table FS 1000-2
Construction Material Selection for Equipment and Sample Containers

Analyte Group	Acceptable Materials
Extractable Organics	Teflon Stainless steel Glass Polypropylene (All types) Polyethylene (All types) All parts of the system including connectors and gaskets must be considered – Viton may be used if no other material is acceptable.
Volatile Organics	Teflon Stainless steel Glass Polypropylene (All types) Polyethylene (All types) All parts of the system including connectors and gaskets must be considered – Viton may be used if no other material is acceptable.
Metals	Teflon Stainless steel Polyethylene (All types) Polypropylene (All types) Tygon, Viton, Silicone, Neoprene PVC Glass (except silica and boron)
Ultratrace Metals	Teflon Polyethylene (All types) Polypropylene (All types) Polycarbonate Mercury must be in glass or Teflon
Inorganic Nonmetallics	Teflon Stainless steel Low carbon, Galvanized or Carbon steel Polyethylene (All types) Polypropylene (All types) Tygon, Viton, Silicone, Neoprene PVC Glass Brass

Table FS 1000-2
Construction Material Selection for Equipment and Sample Containers

Analyte Group	Acceptable Materials
Microbiological samples	Teflon Stainless steel Polyethylene (All types) Polypropylene (All types) Tygon, Viton, Silicone, Neoprene PVC Glass Sterilize all **sample** containers. Thoroughly clean **sampling equipment** and rinse several times with sample water before collection. Sampling equipment **does not require** sterilization **Do not rinse sample containers**

Table FS 1000-3
Equipment Use and Construction

EQUIPMENT	CONSTRUCTION HOUSING[1]	CONSTRUCTION TUBING	USE	PERMISSIBLE ANALYTE GROUPS	RESTRICTIONS AND PRECAUTIONS
WATER SAMPLING					
GROUNDWATER					
1 Positive displacement pumps[2]					
a. Submersible (turbine, helical rotor, gear driven)	SS, Teflon	SS, Teflon, PE, PP	Purging	All analyte groups	[3,4;5]; must be variable speed
			Sampling	All analyte groups	[3,4,5] must be variable speed
	SS, Teflon	Non-inert[6]	Purging	All analyte groups	[3,4,5] must be variable speed; polishing required[7]
			Sampling	All analyte groups <u>except</u> volatile and extractable organics	Must be variable speed If sampling for metals, the tubing must be non-metallic if not SS
	Non-inert[6]	Non-inert[6]	Purging	All analyte groups	[3,4,5] must be variable speed; polishing required[7]
			Sampling	All analyte groups <u>except</u> volatile and extractable organics	Must be variable speed If sampling for metals, the tubing must be non-metallic if not SS
b. Bladder pump (no gas contact)	SS, Teflon, PE, PP or PVC if permanently installed	SS, Teflon, PE, PP	Purging	All analyte groups	[3,4,5] must be variable speed
			Sampling	All analyte groups	[3,4] must be variable speed Bladder must be Teflon if sampling for volatile or extractable organics or PE or PP if used in portable pumps
	SS, Teflon, PE, PP	Non-inert[6]	Purging	All analyte groups	[3,4] must be variable speed; polishing required[7] **This configuration <u>is</u> <u>not</u> recommended**
			Sampling	All analyte groups <u>except</u> volatile and extractable organics	[3,4] must be variable speed If sampling for metals, the tubing must be non-metallic if not SS
	Non-inert[6]	Non-inert[6]	Purging	All analyte groups	[3,4] must be variable speed; polishing required[7]
			Sampling	All analyte groups <u>except</u> volatile and extractable organics	[3,4] must be variable speed; polishing required[7] If sampling for metals, the tubing must be non-metallic if not SS

Table FS 1000-3
Equipment Use and Construction

EQUIPMENT	CONSTRUCTION HOUSING[1]	TUBING	USE	PERMISSIBLE ANALYTE GROUPS	RESTRICTIONS AND PRECAUTIONS
2. Suction lift pumps					
a. Centrifugal	N/A	SS, Teflon, PE, PP	Purging	All analyte groups	[4] foot-valve required Must be variable speed
	N/A	Non-inert[6]	Purging	All analyte groups	[4] foot-valve required; polishing required[7] Must be variable speed
b. Peristaltic	N/A	SS, Teflon, PE, PP	Purging	All analyte groups	[4] foot-valve required; polishing required[7] or continuous pumping required Must be variable speed
			Sampling	All analyte groups except volatile organics	[4] Silicone tubing in pump head Must be variable speed
	N/A	Non-inert[6]	Purging	All analyte groups	[4] foot-valve required Must be variable speed
			Sampling	All analyte groups except volatile and extractable organics	[4] Silicone tubing in pump head Must be variable speed
3. Bailers	SS, Teflon, PE, PP	N/A	Purging	All analyte groups	None; **not recommended**
		N/A	Sampling	All analyte groups	None; **not recommended**
	Non-inert[6]	N/A	Purging	All analyte groups except volatile and extractable organics	None; **not recommended** If sampling for metals, the tubing must be non-metallic if not SS
			Sampling	All analyte groups except volatile and extractable organics	None; **not recommended** If sampling for metals, the tubing must be non-metallic if not SS
SURFACE WATER					
1. Intermediate containers such as pond sampler, scoops, beakers, buckets, and dippers	SS, Teflon, Teflon-coated, PE, PP	N/A	Grab sampling	All analyte groups	None
	Glass	N/A		All analyte groups except boron and fluoride	None
	Non-inert[6]	N/A		All analyte groups except volatile and extractable organics	None

Table FS 1000-3
Equipment Use and Construction

EQUIPMENT	CONSTRUCTION HOUSING[1]	TUBING	USE	PERMISSIBLE ANALYTE GROUPS	RESTRICTIONS AND PRECAUTIONS
2. Nansen, Kemmerer, Van Dorn, Alpha and Beta Samplers, Niskin (or equivalent)	SS, Teflon, Teflon-coated, PE, PP	N/A	Specific depth grab sampling	All analyte groups	None
	Non-inert[6]	N/A		All analyte groups <u>except</u> volatile and extractable organics	None
3. DO Dunker	SS, Teflon, glass, PE, PP	N/A	Water column composite sampling	All analyte groups	None
4 Bailers – double valve	SS, Teflon, PE, PP	N/A	Grab sampling	All analyte groups	None
	Non-inert[6]	N/A	Grab sampling	All analyte groups <u>except</u> volatile and extractable organics	None If sampling for metals, the tubing must be non-metallic if not SS
5. Peristaltic pump	N/A	SS, Teflon, PE, PP	Specific depth sampling	All analyte groups <u>except</u> volatile organics	Silicone tubing in pump head Must be variable speed
	N/A	Non-inert[6]		All analyte groups <u>except</u> volatile and extractable organics	Silicone tubing in pump head Must be variable speed
<u>**FIELD FILTRATION UNITS**</u>	N/A		Dissolved constituents	Inorganic nonmetallics and metals in surface water	Must use a 0.45 μm filter
				Inorganic nonmetallics in groundwater	Must use a 0.45 μm filter
				Metals in groundwater and static wastewater and surface water	Must use in-line, high capacity, one-piece molded filter that is connected to the outlet of a pump; no intermediate vessels; positive pressure PE, PP & Teflon bailers acceptable Must use a 1 μm filter in groundwater, a 0.45 μm filter in surface water
				Metals in moving surface water (i.e., river/stream)	Must use positive pressure device, but an intermediate vessel may be used. Use a 0.45 μm filter

Table FS 1000-3
Equipment Use and Construction

EQUIPMENT	CONSTRUCTION HOUSING[1]	TUBING	USE	PERMISSIBLE ANALYTE GROUPS	RESTRICTIONS AND PRECAUTIONS
SOLID SAMPLING					
SOILS					
1. Core barrel (or liner)	SS, Teflon, glass, Teflon-coated, aluminum, PE, PP	N/A	Sampling	All analyte groups[8]	[9, 10, 11]
	Non-inert[6] nonmetallics	N/A	Sampling	All analyte groups	[12]
	Non-inert[6] metals	N/A	Sampling	All analyte groups	[12]
2. Trowel, scoop, spoon or spatula	SS, Teflon, Teflon-coated, PE, PP	N/A	Sampling	All analyte groups[8]	
			Compositing	All analyte groups except volatile organics	Samples for volatile organics must grab samples
	Plastic	N/A	Sampling and compositing	All analyte groups <u>except</u> volatile and extractable organics	None Must be nonmetallic if not SS
3. Mixing tray (pan)	SS, Teflon, glass, Teflon-coated, aluminum , PE, PP	N/A	Sampling	All analyte groups[8]	[11]
			Compositing or homogenizing	All analyte groups except volatile organics	[11]
	Non-inert[6]	N/A	Compositing or homogenizing	All analyte groups	[10,11,12] must be nonmetallic if not SS
4. Shovel, bucket auger	SS	N/A	Sampling	All analyte groups[8]	None
	Non-SS	N/A	Sampling	All analyte groups[8]	[10,11,12]
5. Split spoon	SS or carbon steel w/ Teflon insert	N/A	Sampling	All analyte groups[8]	[10,11,12]
6. Shelby tube	SS	N/A	Sampling	All analyte groups[8]	[9]
	Carbon steel	N/A	Sampling	All analyte groups	[9,10,12]
SEDIMENT					
1. Coring devices	SS, Teflon, glass, Teflon-coated, aluminum, PE, PP	N/A	Sampling	All analyte groups[8]	[9,10,11]

Table FS 1000-3
Equipment Use and Construction

EQUIPMENT	CONSTRUCTION HOUSING[1]	TUBING	USE	PERMISSIBLE ANALYTE GROUPS	RESTRICTIONS AND PRECAUTIONS
	Non-inert[6] nonmetallics	N/A	Sampling	All analyte groups	[12]
	Non-inert[6] metals	N/A	Sampling	All analyte groups	[9,10,11]
2. Grab – Young, Petersen, Shipek	Teflon, Teflon-lined, SS	N/A	Sampling	All analyte groups[8]	None
	Carbon steel	N/A	Sampling	All analyte groups	[10,11]
3. Dredges – Eckman, Ponar, Petit Ponar Van Veen	SS	N/A	Sampling	All analyte groups[8]	None
	Carbon steel, brass	N/A	Sampling	All analyte groups	[10,11]
4. Trowel, scoop, spoon or spatula	SS, Teflon, Teflon-coated, PE, PP	N/A	Sampling	All analyte groups[8]	
			Compositing	All analyte groups except volatile organics	Samples for volatile organics be grab samples
	Plastic	N/A	Sampling and compositing	All analyte groups except volatile and extractable organics	None must be nonmetallic if not SS
5. Mixing tray (pan)	SS, Teflon, glass, Teflon-coated, aluminum, PE, PP	N/A	Sampling	All analyte groups[8]	[11]
			Compositing or homogenizing	All analyte groups except volatile organics	[11]
	Non-inert[6]	N/A	Compositing or homogenizing	All analyte groups except volatile and extractable organics	none [11] must be nonmetallic if not SS
WASTE[13]					
Scoop	SS	N/A	Liquids, solids & sludges	All analyte groups[8]	Cannot collect deeper phases
Spoon	SS	N/A	Solids, sludges	All analyte groups[8]	Cannot collect deeper phases
Push tube	SS	N/A	Solids, sludges	All analyte groups[8]	Cannot collect deeper phases
Auger	SS	N/A	Solids	All analyte groups[8]	None

Table FS 1000-3
Equipment Use and Construction

EQUIPMENT	CONSTRUCTION HOUSING[1]	CONSTRUCTION TUBING	USE	PERMISSIBLE ANALYTE GROUPS	RESTRICTIONS AND PRECAUTIONS
Sediment sampler	SS	N/A	Impoundments, piles	All analyte groups[8]	None
Ponar dredge	SS	N/A	Solids, sludges & sediments	All analyte groups[8]	None
Coliwasa, Drum thief	Glass	N/A	Liquids, sludges	All analyte groups	None
Mucksucker, Dipstick	Teflon		Liquids, sludges	All analyte groups	Not recommended for tanks > 11 feet deep
Bacon bomb	SS	N/A	Liquids	All analyte groups[8]	Not recommended for viscous wastes
Bailer	SS, Teflon	N/A	Liquids	All analyte groups[8]	Do not use with heterogeneous wastes Not recommended for viscous wastes
Peristaltic pump	N/A	Teflon, Glass	Liquids	All analyte groups except volatile organics	Do not use in flammable atmosphere Not recommended for viscous wastes
Backhoe bucket	Steel	N/A	Solids, Sludges		Difficult to clean Volatiles and metals must be taken from the interior part of the sample
Split spoon	SS	N/A	Solids	All analyte groups[8]	
Roto-Hammer	Steel	N/A	Solids	All analyte groups[8]	Physically breaks up sample Not for flammable atmospheres

Acronyms:

N/A	not applicable
SS	stainless steel
HDPE	high-density polyethylene
PE	polyethylene
PVC	polyvinyl chloride
PP	polypropylene

Table FS 1000-3
Equipment Use and Construction

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[1] Refers to tubing and pump housings/internal parts that are in contact with purged or sampled water (interior and exterior of delivery tube, inner lining of the discharge tube, etc.).

[2] If used to collect volatile or extractable organics, all power cords and other tubing must be encased in Teflon, PE or PP.

[3] If used as a non-dedicated system, pump must be completely disassembled, if practical, and cleaned between wells.

[4] Delivery tubing must be precleaned and precut at the base of operations or laboratory. If the same tubing is used during the sampling event, it must be cleaned and decontaminated between uses.

[5] In-line check valve required.

[6] "Non-inert" pertains to materials that are reactive (adsorb, absorb, etc.) to the analytes being sampled. For organics, materials include rubber, plastics (except PE and PP), and PVC. For metals, materials include brass, galvanized, and carbon steel.

[7] "Polishing": When purging for volatile or extractable organics, the entire length of tubing or the portion which comes in contact with the formation water must be constructed of Teflon, SS, PE or PP. If other materials (e.g., PVC, garden hoses, etc.) are used, the following protocols must be followed: 1) slowly withdraw the pump from the water column during the last phase of purging, to remove any water from the well that may have contacted the exterior of the pump and/or tubing; 2) remove a single well volume with the sampling device before sampling begins. **Do not use Tygon** for purging if purgeable or extractable organics are of interest. Polishing **is not recommended;** use of sampling equipment constructed of appropriate materials is preferred.

[8] Do not use if collecting for hexavalent chromium (Chromium^{+6})

[9] If samples are sealed in the liner for transport to the laboratory, the sample for VOC analysis must be taken from the interior part of the core.

[10] If a non-stainless steel (carbon steel, aluminum) liner, core barrel or implement is used, take the samples for metals, purgeable organics and organics from the interior part of the core sample.

[11] Aluminum foil, trays or liners may be used only if aluminum is not an analyte of interest.

[12] If non-inert-liner, core barrel or implement is used, take samples from the interior part of the collected sample.

[13] If disposable equipment of alternative construction materials is used, the construction material must be compatible with the chemical composition of the waste, cannot alter the characteristics of the waste sample in any way, and cannot contribute analytes of interest or any interfering components.

Table FS1000-4

40 CFR Part 136 TABLE II: Required Containers, Preservation Techniques, and Holding Times

Applicable to **all** Non-Potable Water Samples (includes wastewater, surface water, and groundwater)

Parameter No./Name (refers to parameter number on Tables IA,B, C, D,E, F, G & H as noted)	Container[1]	Preservation[2, 3]	Maximum holding time[4]
Table IA—Bacterial Tests:			
1–5. Coliform, total, fecal, and *E. coli*	PA, G	Cool, <10 °C, 0.0008% $Na_2S_2O_3$ [5]	6 hours[6,7]
6. Fecal streptococci	PA, G	Cool, <10 °C, 0.0008% $Na_2S_2O_3$ [5]	6 hours[6]
7. Enterococci	PA, G	Cool, <10 °C, 0.0008% $Na_2S_2O_3$ [5]	6 hours[6]
8. Salmonella	PA, G	Cool, <10 °C, 0.0008% $Na_2S_2O_3$ [5]	6 hours[6]
Table IA— Aquatic Toxicity Tests:			
9–11. Toxicity, acute and chronic	P, FP, G	Cool, ≤6 °C [8]	36 hours
Table IB—Inorganic Tests:			
1. Acidity	P, FP, G	Cool, ≤6 °C[9]	14 days
2. Alkalinity	P, FP, G	Cool, ≤6 °C[9]	14 days
4. Ammonia	P, FP, G	Cool, ≤6 °C[9], H_2SO_4 to pH<2	28 days
9. Biochemical oxygen demand	P, FP, G	Cool, ≤6 °C[9]	48 hours
10. Boron	P, FP, or Quartz	HNO_3 to pH<2	6 months
11. Bromide	P, FP, G	None required	28 days
14. Biochemical oxygen demand, carbonaceous	P, FP G	Cool, ≤6 °C[9]	48 hours
15. Chemical oxygen demand	P, FP, G	Cool, ≤6 °C[9], H_2SO_4 to pH<2	28 days
16. Chloride	P, FP, G	None required	28 days
17. Chlorine, total residual	P, G	None required	Analyze within 15 minutes
21. Color	P, FP, G	Cool, ≤6 °C[9]	48 hours
23–24. Cyanide, total or available (or CATC)	P, FP, G	Cool, ≤6 °C[9], NaOH to pH>12[10], reducing agent [5]	14 days
25. Fluoride	P	None required	28 days
27. Hardness	P, FP, G	HNO_3 or H_2SO_4 to pH<2	6 months
28. Hydrogen ion (pH)	P, FP, G	None required	Analyze within 15 minutes
31, 43. Kjeldahl and organic N	P, FP, G	Cool, ≤6 °C[9], H_2SO_4 to pH<2	28 days
Table IB—Metals:			
7 18. Chromium VI	P, FP, G	Cool, ≤6 °C[9], pH = 9.3–9.7[12]	28 days
35. Mercury (CVAA)	P, FP, G	HNO_3 to pH<2	28 days

Table FS1000-4

40 CFR Part 136 TABLE II: Required Containers, Preservation Techniques, and Holding Times

Applicable to **all** Non-Potable Water Samples (includes wastewater, surface water, and groundwater)

Parameter No./Name (refers to parameter number on Tables IA,B, C, D,E, F, G & H as noted)	Container[1]	Preservation[2, 3]	Maximum holding time[4]
35. Mercury (CVAFS)	FP, G; and FP-lined cap[13]	5 mL/L 12N HCl or 5 mL/L BrCl[13]	90 days[13]
3, 5–8, 12, 13, 19, 20, 22, 26, 29, 30, 32–34, 36, 37, 45, 47, 51, 52, 58–60, 62, 63, 70– 72, 74, 75. Metals, except boron, chromium VI, and mercury.	P, FP, G	HNO_3 to pH<2, or at least 24 hours prior to analysis[14]	6 months
38. Nitrate	P, FP, G	Cool, ≤6 °C[9]	48 hours
39. Nitrate-nitrite	P, FP, G	Cool, ≤6 °C[9], H_2SO_4 to pH<2	28 days
40. Nitrite	P, FP, G	Cool, ≤6 °C[9]	48 hours
41. Oil and grease	G	Cool, ≤6 °C[9], HCl or H2SO4 to pH<2	28 days
42. Organic Carbon	P, FP, G	Cool, ≤6 °C[9], HCl, H_2SO_4, or H_3PO_4 to pH<2.	28 days
44. Orthophosphate	P, FP, G	Cool, ≤6 °C[9]	Filter within 15 minutes; Analyze within 48 hours
46. Oxygen, Dissolved Probe	G, Bottle and top	None required	Analyze within 15 minutes
47. Winkler	G, Bottle and top	Fix on site and store in dark	8 hours
48. Phenols	G	Cool, ≤6 °C[9], H_2SO_4 to pH<2	28 days
49. Phosphorous (elemental)	G	Cool, ≤6 °C[9]	48 hours
50. Phosphorous, total	P, FP, G	Cool, ≤6 °C[9], H_2SO_4 to pH<2	28 days
53. Residue, total	P, FP, G	Cool, ≤6 °C[9]	7 days
54. Residue, Filterable	P, FP, G	Cool, ≤6 °C[9]	7 days
55. Residue, Nonfilterable (TSS)	P, FP, G	Cool, ≤6 °C[9]	7 days
56. Residue, Settleable	P, FP, G	Cool, ≤6 °C[9]	48 hours
57. Residue, Volatile	P, FP, G	Cool, ≤6 °C[9]	7 days
61. Silica	P or Quartz	Cool, ≤6 °C[9]	28 days
64. Specific conductance	P, FP, G	Cool, ≤6 °C[9]	28 days
65. Sulfate	P, FP, G	Cool, ≤6 °C[9]	28 days
66. Sulfide	P, FP, G	Cool, ≤6 °C[9], add zinc acetate plus sodium hydroxide to pH>9	7 days
67. Sulfite	P, FP, G	None required	Analyze within 15 minutes
68. Surfactants	P, FP, G	Cool, ≤6 °C[9]	48 hours

Table FS1000-4

40 CFR Part 136 TABLE II: Required Containers, Preservation Techniques, and Holding Times

Applicable to **all** Non-Potable Water Samples (includes wastewater, surface water, and groundwater)

Parameter No./Name (refers to parameter number on Tables IA,B, C, D,E, F, G & H as noted)	Container[1]	Preservation[2, 3]	Maximum holding time[4]
69. Temperature	P, FP, G	None required	Analyze
73. Turbidity	P, FP, G	Cool, ≤6 °C[9]	48 hours

Table IC—Organic Tests 8			
13, 18–20, 22, 24–28, 34–37, 39–43, 45–47, 56, 76, 104, 105, 108–111, 113. Purgeable Halocarbons	G, FP-lined septum	Cool, ≤6 °C[9], 0.008% $Na_2S_2O_3$ [5]	14 days
6, 57, 106. Purgeable aromatic hydrocarbons	G, FP-lined septum	Cool, ≤6 °C[9], 0.008% $Na_2S_2O_3$ [5], HCl to pH 2[16]	14 days[16]
3, 4. Acrolein and acrylonitrile	G, FP-lined septum	Cool, ≤6 °C[9], 0.008% $Na_2S_2O_3$ [5], pH to 4–5[17]	14 days[17]
23, 30, 44, 49, 53, 77, 80, 81, 98, 100, 112. Phenols[18]	G, FP-lined cap	Cool, ≤6 °C[9], 0.008% $Na_2S_2O_3$ [5]	7 days until extraction, 40 days after extraction
7, 38. Benzidines [18, 19]	G, FP-lined cap	Cool, ≤6 °C[9], 0.008% $Na_2S_2O_3$ [5]	7 days until extraction[20]
14, 17, 48, 50–52. Phthalate esters [18]	G, FP-lined cap	Cool, ≤6 °C[9]	7 days until extraction, 40 days after extraction
82–84. Nitrosamines [18, 21]	G, FP-lined cap	Cool, ≤6 °C[9], store in dark, 0.008% $Na_2S_2O_3$ [5]	7 days until extraction, 40 days after extraction
88–94. PCBs[18]	G, FP-lined cap	Cool, ≤6 °C[9]	1 year until extraction, 1 year after extraction
54, 55, 75, 79. Nitroaromatics and isophorone[18]	G, FP-lined cap	Cool, ≤6 °C[9], store in dark, 0.008% $Na_2S_2O_3$ [5]	7 days until extraction, 40 days after extraction
1, 2, 5, 8–12, 32, 33, 58, 59, 74, 78, 99, 101. Polynuclear aromatic hydrocarbons[18]	G, FP-lined cap	Cool, ≤6 °C[9], store in dark, 0.008% $Na_2S_2O_3$ [5]	7 days until extraction, 40 days after extraction
15, 16, 21, 31, 87. Haloethers[18]	G, FP-lined cap	Cool, ≤6 °C[9], 0.008% $Na_2S_2O_3$ [5]	7 days until extraction, 40 days after extraction
29, 35–37, 63–65, 107. Chlorinated hydrocarbons[18]	G, FP-lined cap	Cool, ≤6 °C[9]	7 days until extraction, 40 days after extraction
60–62, 66–72, 85, 86, 95–97, 102, 103. CDDs/CDFs[18]			
Aqueous Samples: Field and Lab Preservation	G	Cool, ≤6 °C[9], 0.008% $Na_2S_2O_3$ [5], pH<9	1 year

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Applicable to **all** Non-Potable Water Samples (includes wastewater, surface water, and groundwater)

Parameter No./Name (refers to parameter number on Tables IA,B, C, D,E, F, G & H as noted)	Container[1]	Preservation[2, 3]	Maximum holding time[4]
Solids and Mixed-Phase Samples: Field Preservation	G	Cool, ≤6 °C[9]	7 days
Tissue Samples: Field Preservation	G	Cool, ≤6 °C[9]	24 hours
Solids, Mixed-Phase, and Tissue Samples: Lab Preservation	G	Freeze, ≤-10 °C	1 year
Table ID—Pesticides			
Tests: 1–70. Pesticides[18]	G, FP-lined cap	Cool, ≤6 °C[9], pH 5–9[22]	7 days until extraction, 40 days after extraction
Table IE—Radiological Tests:			
1–5. Alpha, beta, and radium	P, FP, G	HNO_3 to pH<2	6 months
Table IH—Bacterial Tests:			
1. *E. coli*			
2. Enterococci	PA, G	Cool, <10 °C, 0.008% $Na_2S_2O_3$ [5]	6 hours[6]
Table IH—Protozoan Tests:			
8. Cryptosporidium	LDPE; field filtration	0–8 °C	96 hours.[23]
9. Giardia	LDPE; field filtration	0–8 °C	96 hours[23]

Reference: This table is adapted from Table II, 40 CFR Part 136, 2007

[1] ''P'' is polyethylene; ''FP'' is fluoropolymer (polytetrafluoroethylene (PTFE; Teflon®), or other fluoropolymer, unless stated otherwise in this Table II; ''G'' is glass; ''PA'' is any plastic that is made of a sterlizable material (polypropylene or other autoclavable plastic); ''LDPE'' is low density polyethylene.

[2] Except where noted in this Table II and the method for the parameter, preserve each grab sample within 15 minutes of collection. For a composite sample collected with an automated sampler (e.g., using a 24-hour composite sampler; see 40 CFR 122.21(g)(7)(i) or 40 CFR Part 403, Appendix E), refrigerate the sample at ≤6 °C during collection unless specified otherwise in this Table II or in the method(s). For a composite sample to be split into separate aliquots for preservation and/or analysis, maintain the sample at ≤6 °C, unless specified otherwise in this Table II or in the method(s), until collection, splitting, and preservation is completed. Add the preservative to the sample container prior to sample collection when the preservative will not compromise the integrity of a grab sample, a composite sample, or an aliquot split from a composite sample; otherwise, preserve the grab sample, composite sample,

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or aliquot split from a composite sample within 15 minutes of collection. If a composite measurement is required but a composite sample would compromise sample integrity, individual grab samples must be collected at prescribed time intervals (e.g., 4 samples over the course of a day, at 6-hour intervals). Grab samples must be analyzed separately and the concentrations averaged. Alternatively, grab samples may be collected in the field and composited in the laboratory if the compositing procedure produces results equivalent to results produced by arithmetic averaging of the results of analysis of individual grab samples. For examples of laboratory compositing procedures, see EPA Method 1664A (oil and grease) and the procedures at 40 CFR 141.34(f)(14)(iv) and (v) (volatile organics).

[3] When any sample is to be shipped by common carrier or sent via the U.S. Postal Service, it must comply with the Department of Transportation Hazardous Materials Regulations (49 CFR Part 172). The person offering such material for transportation is responsible for ensuring such compliance. For the preservation requirements of Table II, the Office of Hazardous Materials, Materials Transportation Bureau, Department of Transportation has determined that the Hazardous Materials Regulations do not apply to the following materials: Hydrochloric acid (HCl) in water solutions at concentrations of 0.04% by weight or less (pH about 1.96 or greater); Nitric acid (HNO3) in water solutions at concentrations of 0.15% by weight or less (pH about 1.62 or greater); Sulfuric acid (H2SO4) in water solutions at concentrations of 0.35% by weight or less (pH about 1.15 or greater); and Sodium hydroxide (NaOH) in water solutions at concentrations of 0.080% by weight or less (pH about 12.30 or less).

[4] Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before the start of analysis and still be considered valid (e.g., samples analyzed for fecal coliforms may be held up to 6 hours prior to commencing analysis). Samples may be held for longer periods only if the permittee or monitoring laboratory has data on file to show that, for the specific types of samples under study, the analytes are stable for the longer time, and has received a variance from the Regional Administrator under § 136.3(e). For a grab sample, the holding time begins at the time of collection. For a composite sample collected with an automated sampler (e.g., using a 24-hour composite sampler; see 40 CFR 122.21(g)(7)(i) or 40 CFR Part 403, Appendix E), the holding time begins at the time of the end of collection of the composite sample. For a set of grab samples composited in the field or laboratory, the holding time begins at the time of collection of the last grab sample in the set. Some samples may not be stable for the maximum time period given in the table. A permittee or monitoring laboratory is obligated to hold the sample for a shorter time if it knows that a shorter time is necessary to maintain sample stability. See § 136.3(e) for details. The date and time of collection of an individual grab sample is the date and time at which the sample is collected. For a set of grab samples to be composited, and that are all collected on the same calendar date, the date of collection is the date on which the samples are collected. For a set of grab samples to be composited, and that are collected across two calendar dates, the date of collection is the dates of the two days; e.g., November 14–15. For a composite sample collected automatically on a given date, the

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date of collection is the date on which the sample is collected. For a composite sample collected automatically, and that is collected across two calendar dates, the date of collection is the dates of the two days; e.g., November 14–15.

[5] Add a reducing agent only if an oxidant (e.g., chlorine) is present. Reducing agents shown to be effective are sodium thiosulfate ($Na_2S_2O_3$), ascorbic acid, sodium arsenite ($NaAsO_2$), or sodium borohydride ($NaBH_4$). However, some of these agents have been shown to produce a positive or negative cyanide bias, depending on other substances in the sample and the analytical method used. Therefore, do not add an excess of reducing agent. Methods recommending ascorbic acid (e.g., EPA Method 335.4) specify adding ascorbic acid crystals, 0.1–0.6 g, until a drop of sample produces no color on potassium iodide (KI) starch paper, then adding 0.06 g (60 mg) for each liter of sample volume. If $NaBH_4$ or $NaAsO_2$ is used, 25 mg/L $NaBH_4$ or 100 mg/L $NaAsO_2$ will reduce more than 50 mg/L of chlorine (see method ‘‘Kelada-01’’ and/or Standard Method

4500–CN^- for more information). After adding reducing agent, test the sample using KI paper, a test strip (e.g. for chlorine, SenSafeTM Total Chlorine Water Check 480010) moistened with acetate buffer solution (see Standard Method 4500–Cl.C.3e), or a chlorine/oxidant test method (e.g., EPA Method 330.4 or 330.5), to make sure all oxidant is removed. If oxidant remains, add more reducing agent. Whatever agent is used, it should be tested to assure that cyanide results are not affected adversely.

[6] Samples analysis should begin immediately, preferably within 2 hours of collection. The maximum transport time to the laboratory is 6 hours, and samples should be processed within 2 hours of receipt at the laboratory.

[7] For fecal coliform samples for sewage sludge (biosolids) only, the holding time is extended to 24 hours for the following sample types using either EPA Method 1680 (LTB–EC) or 1681 (A–1): Class A composted, Class B aerobically digested, and Class B anaerobically digested.

[8] Sufficient ice should be placed with the samples in the shipping container to ensure that ice is still present when the samples arrive at the laboratory. However, even if ice is present when the samples arrive, it is necessary to immediately measure the temperature of the samples and confirm that the preservation temperature maximum has not been exceeded. In the isolated cases where it can be documented that this holding temperature cannot be met, the permittee can be given the option of on-site testing or can request a variance. The request for a variance should include supportive data which show that the toxicity of the effluent samples is not reduced because of the increased holding temperature.

[9] Aqueous samples must be preserved at ≤6 °C, and should not be frozen unless data demonstrating that sample freezing does not adversely impact sample integrity is maintained on file and accepted as valid by the regulatory authority. Also, for purposes of NPDES monitoring, the specification of ‘‘≤°C’’ is used in place of the ‘‘4 °C’’ and ‘‘< 4 °C’’ sample temperature requirements listed in some methods. It is not necessary to measure the sample temperature to three significant figures (1/100th of 1 degree); rather, three

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Applicable to **all** Non-Potable Water Samples (includes wastewater, surface water, and groundwater)

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significant figures are specified so that rounding down to 6 °C may not be used to meet the ≤6 °C requirement. The preservation temperature does not apply to samples that are analyzed immediately (less than 15 minutes).

[10] Sample collection and preservation: Collect a volume of sample appropriate to the analytical method in a bottle of the material specified. If the sample can be analyzed within 48 hours and sulfide is not present, adjust the pH to > 12 with sodium hydroxide solution (e.g., 5% w/v), refrigerate as specified, and analyze within 48 hours. Otherwise, to extend the holding time to 14 days and mitigate interferences, treat the sample immediately using any or all of the following techniques, as necessary, followed by adjustment of the sample pH to > 12 and refrigeration as specified. There may be interferences that are not mitigated by approved procedures. Any procedure for removal or suppression of an interference may be employed, provided the laboratory demonstrates that it more accurately measures cyanide. Particulate cyanide (e.g., ferric ferrocyanide) or a strong cyanide complex (e.g., cobalt cyanide) are more accurately measured if the laboratory holds the sample at room temperature and pH > 12 for a minimum of 4 hours prior to analysis, and performs UV digestion or dissolution under alkaline (pH=12) conditions, if necessary.

(1) SULFUR: To remove elemental sulfur (S8), filter the sample immediately. If the filtration time will exceed 15 minutes, use a larger filter or a method that requires a smaller sample volume (e.g., EPA Method 335.4 or Lachat Method 01). Adjust the pH of the filtrate to > 12 with NaOH, refrigerate the filter and filtrate, and ship or transport to the laboratory. In the laboratory, extract the filter with 100 mL of 5% NaOH solution for a minimum of 2 hours. Filter the extract and discard the solids. Combine the 5% NaOH-extracted filtrate with the initial filtrate, lower the pH to approximately 12 with concentrated hydrochloric or sulfuric acid, and analyze the combined filtrate. Because the detection limit for cyanide will be increased by dilution by the filtrate from the solids, test the sample with and without the solids procedure if a low detection limit for cyanide is necessary. Do not use the solids procedure if a higher cyanide concentration is obtained without it. Alternatively, analyze the filtrates from the sample and the solids separately, add the amounts determined (in µg or mg), and divide by the original sample volume to obtain the cyanide concentration.

(2) SULFIDE: If the sample contains sulfide as determined by lead acetate paper, or if sulfide is known or suspected to be present, immediately conduct one of the volatilization treatments or the precipitation treatment as follows: Volatilization—Headspace expelling. In a fume hood or well-ventilated area, transfer 0.75 liter of sample to a 4.4 L collapsible container (e.g., CubitainerTM). Acidify with concentrated hydrochloric acid to pH < 2. Cap the container and shake vigorously for 30 seconds. Remove the cap and expel the headspace into the fume hood or open area by collapsing the container without expelling the sample. Refill the headspace by expanding the container. Repeat expelling a total of five headspace volumes. Adjust the pH to > 12, refrigerate, and ship or transport to the laboratory. Scaling to a smaller or larger sample volume must maintain the air to sample volume ratio. A larger volume of air will result in too great a loss of cyanide (> 10%). Dynamic stripping: In a fume hood or well- ventilated area, transfer 0.75 liter of sample to a container of the material specified and acidify with concentrated hydrochloric acid to pH < 2. Using a calibrated air sampling pump or flowmeter, purge the acidified sample into the fume hood or open area through a fritted glass aerator at a flow rate of 2.25 L/min for 4 minutes. Adjust the pH to >

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12, refrigerate, and ship or transport to the laboratory. Scaling to a smaller or larger sample volume must maintain the air to sample volume ratio. A larger volume of air will result in too great a loss of cyanide (> 10%). Precipitation: If the sample contains particulate matter that would be removed by filtration, filter the sample prior to treatment to assure that cyanide associated with the particulate matter is included in the measurement. Ship or transport the filter to the laboratory. In the laboratory, extract the filter with 100 mL of 5% NaOH solution for a minimum of 2 hours. Filter the extract and discard the solids. Combine the 5% NaOH-extracted filtrate with the initial filtrate, lower the pH to approximately 12 with concentrated hydrochloric or sulfuric acid, and analyze the combined filtrate. Because the detection limit for cyanide will be increased by dilution by the filtrate from the solids, test the sample with and without the solids procedure if a low detection limit for cyanide is necessary. Do not use the solids procedure if a higher cyanide concentration is obtained without it. Alternatively, analyze the filtrates from the sample and the solids separately, add the amounts determined (in µg or mg), and divide by the original sample volume to obtain the cyanide concentration. For removal of sulfide by precipitation, raise the pH of the sample to > 12 with NaOH solution, then add approximately 1 mg of powdered cadmium chloride for each mL of sample. For example, add approximately 500 mg to a 500-mL sample. Cap and shake the container to mix. Allow the precipitate to settle and test the sample with lead acetate paper. If necessary, add cadmium chloride but avoid adding an excess. Finally, filter through 0.45 micron filter. Cool the sample as specified and ship or transport the filtrate and filter to the laboratory. In the laboratory, extract the filter with 100 mL of 5% NaOH solution for a minimum of 2 hours. Filter the extract and discard the solids. Combine the 5% NaOH-extracted filtrate with the initial filtrate, lower the pH to approximately 12 with concentrated hydrochloric or sulfuric acid, and analyze the combined filtrate. Because the detection limit for cyanide will be increased by dilution by the filtrate from the solids, test the sample with and without the solids procedure if a low detection limit for cyanide is necessary. Do not use the solids procedure if a higher cyanide concentration is obtained without it. Alternatively, analyze the filtrates from the sample and the solids separately, add the amounts determined (in µg or mg), and divide by the original sample volume to obtain the cyanide concentration. If a ligand-exchange method is used (e.g., ASTM D6888), it may be necessary to increase the ligand-exchange reagent to offset any excess of cadmium chloride.

(3) SULFITE, THIOSULFATE, OR THIOCYANATE: If sulfite, thiosulfate, or thiocyanate is known or suspected to be present, use UV digestion with a glass coil (Method Kelada-01) or ligand exchange (Method OIA–1677) to preclude cyanide loss or positive interference.

(4) ALDEHYDE: If formaldehyde, acetaldehyde, or another water-soluble aldehyde is known or suspected to be present, treat the sample with 20 mL of 3.5% ethylenediamine solution per liter of sample.

(5) CARBONATE: Carbonate interference is evidenced by noticeable effervescence upon acidification in the distillation flask, a reduction in the pH of the absorber solution, and incomplete cyanide spike recovery. When significant carbonate is present, adjust the pH to ≥12 using calcium hydroxide instead of sodium hydroxide. Allow the precipitate to settle and decant or filter the sample prior to analysis (also see Standard Method 4500–CN.B.3.d).

Table FS1000-4

40 CFR Part 136 TABLE II: Required Containers, Preservation Techniques, and Holding Times

Applicable to **all** Non-Potable Water Samples (includes wastewater, surface water, and groundwater)

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(6) CHLORINE, HYPOCHLORITE, OR OTHER OXIDANT: Treat a sample known or suspected to contain chlorine, hypochlorite, or other oxidant as directed in footnote 5.

[11] For dissolved metals, filter grab samples within 15 minutes of collection and before adding preservatives. For a composite sample collected with an automated sampler (e.g., using a 24-hour composite sampler; see 40 CFR 122.21(g)(7)(i) or 40 CFR Part 403, Appendix E), filter the sample within 15 minutes after completion of collection and before adding preservatives. If it is known or suspected that dissolved sample integrity will be compromised during collection of a composite sample collected automatically over time (e.g., by interchange of a metal between dissolved and suspended forms), collect and filter grab samples to be composited (footnote 2) in place of a composite sample collected automatically.

[12] To achieve the 28-day holding time, use the ammonium sulfate buffer solution specified in EPA Method 218.6. The allowance in this footnote supersedes preservation and holding time requirements in the approved hexavalent chromium methods, unless this supersession would compromise the measurement, in which case requirements in the method must be followed.

[13] Samples collected for the determination of trace level mercury (<100 ng/L) using EPA Method 1631 must be collected in tightly-capped fluoropolymer or glass bottles and preserved with BrCl or HCl solution within 48 hours of sample collection. The time to preservation may be extended to 28 days if a sample is oxidized in the sample bottle. A sample collected for dissolved trace level mercury should be filtered in the laboratory within 24 hours of the time of collection. However, if circumstances preclude overnight shipment, the sample should be filtered in a designated clean area in the field in accordance with procedures given in Method 1669. If sample integrity will not be maintained by shipment to and filtration in the laboratory, the sample must be filtered in a designated clean area in the field within the time period necessary to maintain sample integrity. A sample that has been collected for determination of total or dissolved trace level mercury must be analyzed within 90 days of sample collection.

[14] An aqueous sample may be collected and shipped without acid preservation. However, acid must be added at least 24 hours before analysis to dissolve any metals that adsorb to the container walls. If the sample must be analyzed within 24 hours of collection, add the acid immediately (see footnote 2). Soil and sediment samples do not need to be preserved with acid. The allowances in this footnote supersede the preservation and holding time requirements in the approved metals methods.

[15] Guidance applies to samples to be analyzed by GC, LC, or GC/MS for specific compounds.

[16] If the sample is not adjusted to pH 2, then the sample must be analyzed within seven days of sampling.

[17] The pH adjustment is not required if acrolein will not be measured. Samples for acrolein receiving no pH adjustment must be analyzed within 3 days of sampling.

Table FS1000-4

40 CFR Part 136 TABLE II: Required Containers, Preservation Techniques, and Holding Times

Applicable to **all** Non-Potable Water Samples (includes wastewater, surface water, and groundwater)

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[18] When the extractable analytes of concern fall within a single chemical category, the specified preservative and maximum holding times should be observed for optimum safeguard of sample integrity (i.e., use all necessary preservatives and hold for the shortest time listed). When the analytes of concern fall within two or more chemical categories, the sample may be preserved by cooling to ≤6 °C, reducing residual chlorine with 0.008% sodium thiosulfate, storing in the dark, and adjusting the pH to 6–9; samples preserved in this manner may be held for seven days before extraction and for forty days after extraction. Exceptions to this optional preservation and holding time procedure are noted in footnote 5 (regarding the requirement for thiosulfate reduction), and footnotes 19, 20 (regarding the analysis of benzidine).

[19] If 1,2-diphenylhydrazine is likely to be present, adjust the pH of the sample to 4.0 ± 0.2 to prevent rearrangement to benzidine.

[20] Extracts may be stored up to 30 days at < 0 °C.

[21] For the analysis of diphenylnitrosamine, add 0.008% $Na_2S_2O_3$ and adjust pH to 7–10 with NaOH within 24 hours of sampling

[22] The pH adjustment may be performed upon receipt at the laboratory and may be omitted if the samples are extracted within 72 hours of collection. For the analysis of aldrin, add 0.008% $Na_2S_2O_3$.

[23] Holding time is calculated from time of sample collection to elution for samples shipped to the laboratory in bulk and calculated from the time of sample filtration to elution for samples filtered in the field

Table FS 1000-5
Approved Water and Wastewater Procedures, Containers, Preservation and Holding Times
For Analytes not Found in 40 CFR 136

Analyte	Methods	Reference [1]	Container [2]	Preservation [3]	Maximum Holding Time [4]
Bromine	DPD Colorimetric [5]	SM 4500-Cl-G	P, G	None required	Analyze immediately
Bromates	Ion Chromatography	EPA 300.0 [6]	P, G	Cool 4°C	30 days
Chlorophylls	Spectrophotometric	SM 10200 H	P, G [7]	Dark 4°C Filtered, dark, -20°C	48 hours chilled until filtration [8], and analyze immediately or 48 hours chilled until filtration [8], and 28 days (frozen) after filtration
Corrosivity	Calculated ($CaCO_3$ Stability, Langelier Index)	SM 2330 ASTM D513-92	P, G	Cool 4°C [9]	7 days [9]
FL-PRO	Gas Chromatography	DEP (11/1/95)	G only	Cool 4°C, H_2SO_4 or HCl to pH<2	7 days until extraction, 40 days after extraction
Odor	Human Panel	SM 2150	G only	Cool 4°C	6 hours
Salinity	Electrometric [10] Hydrometric [10]	SM 2520 B SM 2520 C	G, wax seal	Analyze immediately or use wax seal	30 days [10]
Taste	Human Panel	SM 2160 B, C, D ASTM E679-91	G only	Cool 4°C	24 hours
Total Dissolved Gases	Direct-sensing Membrane-diffusion	SM 2810	___________	___________	Analyze in-situ
Total Petroleum Hydrocarbons	Gravimetry	EPA 1664	G only	Cool 4°C, H_2SO_4 or HCl to pH<2	28 days
Transparency	Irradiometric [11]	62-302.200(6), FAC	___________	___________	Analyze in-situ
Un-ionized Ammonia	Calculated [12]	DEP-SOP [13]	P, G	Cool 4°C $Na_2S_2O_3$ [12]	8 hours unpreserved 28 days preserved [12]
Organic Pesticides [14]	GC and HPLC	EPA (600-series) [14]	[15]	[15]	[15]

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[1] SM XXXX = procedures from "Standard Methods for the Examination of Water and Wastewater", APHA-AWWA-WPCF, 20th edition, 1998 and Standard Methods Online.

ASTM XXXX-YY = procedure from "Annual Book of ASTM Standards", Volumes 11.01 and 11.02 (Water I and II), 1999.

[2] P = plastic, G = glass.

[3] When specified, sample preservation should be performed immediately upon sample collection.

[4] The times listed are the maximum times that samples may be held before analysis and still be considered valid.

Table FS 1000-5
Approved Water and Wastewater Procedures, Containers, Preservation and Holding Times
For Analytes not Found in 40 CFR 136

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[5] The approved procedure is for residual chlorine. However, in the absence of chlorine, the DPD colorimetric procedure can be adapted to measure bromine content of the sample. In such case, the validity of this assumption must be verified by using another procedure for chlorine which is not affected by the presence of bromine (i.e., negligible interference).

[6] The Determination of Inorganic Anions in Water by Ion Chromatography", EPA Method 300.0, Revised August 1993, by John D. Pfaff, U. S. EPA Cincinnati, Ohio 45268.

[7] Collect samples in opaque bottles and process under reduced light.

[8] Samples must be filtered within 48 hours of collection. Add magnesium carbonate to the filter while the last of the sample passes through the filter..

[9] Temperature and pH must be measured on site at the time of sample collection. 7 days is the maximum time for laboratory analysis of total alkalinity, calcium ion and total solids.

[10] The electrometric and hydrometric analytical methods are suited for field use. The argentometric method is suited for laboratory use. Samples collected for laboratory analysis, when properly sealed with paraffin waxed stopper, may be held indefinitely. The maximum holding time of 30 days is recommended as a practical regulatory limit.

[11] Transparency in surface waters is defined as a compensation point for photosynthetic activity, i.e., the depth at which one percent of the light intensity entering at the water surface remains unabsorbed. The DEP Chapter 62-302, FAC requires that the light intensities at the surface and subsurface be measured simultaneously by irradiance meters such as the Kahlsico Underwater Irradiometer, Model No. 268 WA 310, or an equivalent device having a comparable spectral response.

[12] The results of the measurements of pH, temperature, salinity (if applicable) and the ammonium ion concentration in the sample are used to calculate the concentration of ammonia in the unionized state. Temperature, pH and salinity must be measured on-site at the time of sample collection. Laboratory analysis of the ammonium ion concentration should be conducted within eight hours of sample collection. If prompt analysis of ammonia is impossible, preserve samples with H_2SO_4 to pH between 1.5 and 2. Acid-preserved samples, stored at 4°C, may be held up to 28 days for ammonia determination. Sodium thiosulfate should only be used if fresh samples contain residual chlorine.

[13] DEP Central Analytical Laboratory, Tallahassee, FL, Revision No. 2, 2-12-2001. The document is available from the DEP Standards & Assessment Section..

[14] Other pesticides listed in approved EPA methods (608.1, 608.2, 614, 614.1, 615, 617, 618, 619, 622, 622.1, 627, 629, 631, 632, 632.1, 633, 642, 643, 644 and 645) that are not included in Table ID of 40 CFR Part 136 (July 2007).

[15] Container, preservation and holding time as specified in each individual method must be followed.

Table FS 1000-6
Recommended Sample Containers, Sample Volumes, Preservation Techniques and Holding Times for Residuals, Soil and Sediment Samples

Analyte	Methods	References	Container	Preservation	Maximum Holding Times
Volatile Organics	Purge-and-Trap GC and GC-MS	8015, 8260, 8021, 5035	See Table 1000-7		
Semivolatile Organics	GC, HPLC, and GC-MS	8041, 8061, 8070, 8081, 8082, 8091, 8111, 8121, 8131, 8141, 8151, 8270, 8275, 8280, 8290, 8310, 8315, 8316, 8318, 8321, 8325, 8330, 8331, 8332, 8410, 8430, 8440, FL-PRO	Glass, 8 oz widemouth with Teflon® -Lined lid	Cool 4°C [1]	14 days until extraction, 40 days after extraction
Dioxins		8290	Amber Glass, 8 oz widemouth with Teflon® -Lined lid	Cool 4°C[1] in dark	30 days until extraction, 45 days after extraction
Total Metals-except mercury and chromium VI methods	Flame AA, Furnace AA, Hydride and ICP	All 7000-series (except 7195, 7196, 7197, 7198, 7470 and 7471), and 6010 (ICP)	Glass or plastic 8 oz widemouth (200 grams sample)	None	6 months
Chromium VI	Colorimetric, Chelation with Flame AA (200 gram sample)	7196 and 7197 (prep 3060)	Glass or plastic, 8 oz widemouth (200 gram sample)	Cool 4°± 2°C[1]	1 month until extraction, 4 days after extraction [2]
Mercury	Manual Cold Vapor AA	7471	Glass or plastic 8 oz widemouth (200 grams sample)	Cool 4°± 2°C[1]	28 days
Microbiology (MPN)		MPN	Sterile glass or plastic	Cool 4°C[1]	24 hours
Aggregate Properties			Glass or plastic	Cool 4°C[1]	14 days
Inorganic nonmetallics all except:			Glass or plastic	Cool 4°C[1]	28 days
Sulfite, Nitrate, Nitrite & o-phosphate			Glass or plastic		48 hours
Elemental Phosphorus			Glass		48 hours

Table FS 1000-6

Recommended Sample Containers, Sample Volumes, Preservation Techniques and Holding Times for Residuals, Soil and Sediment Samples

The term “residuals” include: (1) sludges of domestic origin having no specific requirements in Tables FS-1000-4 or FS-1000-9; (2) sludges of industrial origin; and (3) concentrated waste samples.

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[1] Keep soils, sediments and sludges cool at 4°C from collection time until analysis. No preservation is required for concentrated waste samples.

[2] Storage Temperature is 4°C, ±2°C

Table FS 1000-7

Sample Handling, Preservation and Holding Time Table for SW 846 Method 5035

			Sample Container					
Conc. Level	Sampling Device	Collection Procedure	Type	Vial Preparation	Preservation	Sample Preparation	Max HT[1]	Determinative Procedure
≤200 ug/kg	Coring Device	5035 - Section 6.2.1	Glass Vial w/ PTFE-silicone Septum	5035 - 6.1.1	$NaHSO_4$ / 4ºC	5035 - Section 7.2	14 D	Any recognized VOC Method
				5035 - 6.1.1[2]	4ºC	5035 - Section 7.2	48 H	Any recognized VOC Method
				5035 - 6.1.1[2]	4ºC / -10ºC[3],[4]	5035 - Section 7.2	48 H / 14 D[5]	Any recognized VOC Method
	EnCore or equivalent	5035 - Section 6.2.1	EnCore or equivalent	5035 - 6.1.1[2],[6],[7]	4ºC	5035 - Section 7.2	48 H	Any recognized VOC Method
		5035 - Section 6.2.1	EnCore or equivalent	5035 - 6.1.1[6],[7]	$NaHSO_4$ / 4ºC	5035 - Section 7.2[5]	48 H / 14 D[5]	Any recognized VOC Method
		5035 - Section 6.2.1	EnCore or equivalent	5035-6.1.1[2][6][7]	4ºC / -10ºC[3],[4]	5035 - Section 7.2[5]	48 H / 14 D[5]	Any recognized VOC Method
>200 ug/kg	EnCore or equivalent	5035 - Section 6.2.2.3[6]	EnCore or equivalent	5035 - 6.1.3[6],[7]	4ºC	5035 - Sections 7.3.2 & 7.3.3[5]	48 H / 14 D[5]	Any recognized VOC Method
>200 ug/kg[8]	Coring Device	5035 - Section 6.2.2.3[9]	Glass Vial w/ PTFE-silicone Septum	6.1.3[9]	Methanol/PEG + 4ºC	5035 - Section 7.3.4	14 D	Any recognized VOC Method
	Conventional Devices	DEP SOP - Section 4.3	Glass w/ PTFE-silicone Septum	6.1.2	4ºC	5035 - Sections 7.3.1 - 7.3.3	14 D	Any recognized VOC Method
Oily Waste	Conventional Devices	5035 - Section 6.2.4.2	Glass w/ PTFE-silicone Septum	6.1.4	4ºC	5035 - Sections 7.4.1 - 7.4.2	14 D	Any recognized VOC Method
	Conventional Devices	5035 - Section 6.2.4.1	Glass w/ PTFE-silicone Septum	6.1.4	Methanol/PEG + 4ºC	5035 - Sections 7.4.3	14 D	Any recognized VOC Method
Dry Wt.	Conventional Devices		Glass with Teflon liner		4ºC	5035 - Section 7.5		
Soil Screen	Conventional Devices	DEP SOP - Section 4.3	Glass w/ PTFE-silicone Septum		4ºC	5035 - Section 7.1	14 D	Any recognized VOC Method

Table FS 1000-7

Sample Handling, Preservation and Holding Time Table for SW 846 Method 5035

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① Maximum time allowable from time/date of collection to sample analysis.

② Eliminate 6.1.1.2; use <u>only</u> organic-free water.

③ Contents of sampling device must be transported to the laboratory at 4ºC and stored at -10ºC.

④ In order to ensure that vials do not break during freezing, they should be stored on their side or at a slanted angle to maximize surface area.

⑤ Maximum allowable time at 4ºC is 48 hours; maximum allowable time to sample analysis is 14 days (from time of sample collection).

⑥ Conducted in the laboratory.

⑦ Entire contents of sampling device are extruded into the sample analysis vial containing the appropriate solvent.

⑧ Procedures are limited only to those situations or programs in which the maximum contamination level <u>does not exceed</u> 200 ug/kg.

⑨ Methanolic preservation in the field is not recommended, but may be used if approved by an DEP program.

FS 1000-8
Preservation Methods and Holding Times for Drinking Water Samples that Differ from 40 CFR Part 136, Table II

Analyte	Preservation[1]	Holding Time[2]	Holding Time for Extract[3]	Container[4]
MICROBIOLOGICAL-BACTERIA	Cool < 10°C, $Na_2S_2O_3$[5]			P or G
Total Coliforms, fecal coliforms & *E. coli* in drinking water	Cool < 10°C[6], $Na_2S_2O_3$[5]	30 Hours[7]		P or G
Total coliforms and fecal coliforms in source water Heterotrophic bacteria in drinking water	Cool < 10°C, $Na_2S_2O_3$[5]	8 hours		P or G
Gross Alpha	Conc. HCl or HNO_3 to pH <2 [8,9]	6 mo		P or G
Gross beta	Conc. HCl or HNO to pH <2[8,9]	6 mo		P or G
Strontium-89	Conc. HCl or HNO to pH <2[8,9]	6 mo		P or G
Strontium-90	Conc. HCl or HNO to pH <2[8,9]	6 mo		P or G
Radium-226	Conc. HCl or HNO to pH <2[8,9]	6 mo		P or G
Radium-228	Conc. HCl or HNO to pH <2[8,9]	6 mo		P or G
Cesium-134	Concentrated HCl to pH <<2[8,9]	6 mo		P or G
Iodine-131	None	8 da		P or G
Tritium	None	6 months		G
Uranium	Conc. HCl or HNO_3 to pH <2[8,9]	6 mo		P or G
Photon emitters	Conc. HCl or HNO_3 to pH <2[8,9]	6 mo		P or G
Asbestos	Cool 4°C	48 hours		P or G
Bromate	Ethylenediamine (50mg/L)	28 days		P or G
Cyanide	Cool, 4C, Ascorbic acid (if chlorinated), NaOH pH>12	14 days		P or G
Nitrate	Cool, 4°C	48 hours		P or G
Nitrate (chlorinated source)	Cool, 4°C	14 days		P or G
Odor	Cool 4°C	24 hours		G
502.2	Sodium Thiosulfate or Ascorbic Acid, 4°C HCl pH<2 if Ascorbic Acid is used	14 days		Glass with PFTE Lined Septum

FS 1000-8
Preservation Methods and Holding Times for Drinking Water Samples that Differ from 40 CFR Part 136, Table II

Analyte	Preservation[1]	Holding Time[2]	Holding Time for Extract[3]	Container[4]
504.1	Sodium Thiosulfate Cool, 4°C,	14 days	4°C, 24 hours	Glass with PFTE-Lined Septum
505	Sodium Thiosulfate Cool, 4°C	14 days (7 days for Heptachlor)	4°C, 24 hours	Glass with PFTE-Lined Septum
506	Sodium Thiosulfate Cool, 4°C, Dark	14 days	4°C, dark, 14 days	Amber Glass with PFTE-lined Cap
507	Sodium Thiosulfate Cool, 4°C, Dark	14 days (see method for exceptions)	4°C, dark, 14 days	Amber Glass with PFTE-lined Cap
508	Sodium Thiosulfate Cool, 4°C, Dark	7 days (see method for exceptions)	4°C, dark, 14 days	Glass with PFTE-lined Cap
508A	Cool, 4°C	14 days	30 days	Glass with PFTE-lined Cap
508.1	Sodium Sulfite, HCl pH<2, Cool, 4°C	14 days (see method for exceptions)	30 days	Glass with PFTE-lined Cap
515.1	Sodium Thiosulfate Cool, 4°C, Dark	14 days	4°C, dark, 28 days	Amber Glass with PFTE-lined Cap
515.2	Sodium Thiosulfate HCl pH<2, Cool, 4°C, Dark	14 days	≤ 4°C, dark, 14 days	Amber Glass with PFTE-lined Cap
515.3	Sodium Thiosulfate HCl pH<2, Cool, 4°C, Dark	14 days	≤ 4°C, dark, 14 days	Amber Glass with PFTE-lined Cap
515.4	Sodium Sulfite, HCl pH<2, Cool, ≤10°C for first 48 hours ≤6°C thereafter, Dark	14 days	≤0°C, 21 days	
524.2	Ascorbic Acid, HCl pH<2, Cool 4°C	14 days		Glass with PFTE-lined Septum
525.2	Sodium Sulfite, Dark, Cool, 4°C, HCl pH<2	14 days (see method for exceptions)	≤ 4°C, 30 days from collection	Amber Glass with PFTE-lined Cap
531.1, 6610	Sodium Thiosulfate Monochloroacetic acid, pH<3, Cool, 4°C	Cool 4°C, 28 days		Glass with PFTE-lined Septum
531.2	Sodium Thiosulfate, Potassium Dihydrogen Citrate buffer to pH 4,	28 days		

FS 1000-8
Preservation Methods and Holding Times for Drinking Water Samples that Differ from 40 CFR Part 136, Table II

Analyte	Preservation[1]	Holding Time[2]	Holding Time for Extract[3]	Container[4]
	dark, ≤10°C for first 48 hr, ≤6°C thereafter			
547	Sodium Thiosulfate Cool, 4°C	14 days (18 mo. frozen)		Glass with PFTE-lined Septum
548.1	Sodium Thiosulfate (HCl pH 1.5-2 if high biological activity), Cool, 4°C, Dark	7 days	≤4°C 14 days	Amber Glass with PFTE-lined Septum
549.2	Sodium Thiosulfate (H_2SO_4 pH<2 if biologically active), Cool, 4°C, Dark	7 days	21 days	High Density Amber Plastic or Silanized Amber Glass
550, 550.1	Sodium Thiosulfate Cool, 4°C, HCl pH<2	7 days	550, 30 days 550.1, 40 days Dark, 4°C	Amber Glass with PFTE-lined Cap
551.1	Sodium Thiosulfate, Sodium Sulfite, Ammonium Chloride, pH 4.5-5.0 with phosphate buffer, Cool, 4°C	14 days		Glass with PFTE-lined Septum
552.1	Ammonium chloride, Cool, 4°C, Dark	14 days	≤4°C, dark 48 hours	Amber Glass with PFTE-lined cap
552.2	Ammonium chloride, Cool, 4°C, Dark	14 days	≤4°C, dark 7 days ≤-10°C 14 days	Amber Glass with PFTE-lined cap
555	Sodium Sulfite, HCl, pH ≤ 2, Dark, Cool 4°C	14 days		Glass with PFTE-lined cap
1613B	Sodium Thiosulfate, Cool, 0-4°C, Dark		Recommend 40 days	Amber Glass with PFTE-lined Cap

[1] Preservation, when required, must be done immediately upon sample collection.

[2] Stated values are the maximum regulatory holding times. Sample processing must begin by the stated time.

[3] Stated time is the maximum time a prepared sample extract may be held before analysis.

[4] (P) polyethylene or (G) or glass. For microbiology, plastic sample containers must be made of sterilizable materials (poly-propylene or other autoclavable plastic).

[5] Addition of sodium thiosulfate is only required if the sample has a detectable amount of residual chlorine, as indicated by a field test using EPA Method 330.4 or 330.2 or equivalent.

FS 1000-8
Preservation Methods and Holding Times for Drinking Water Samples that Differ from 40 CFR Part 136, Table II

[6] Temperature requirement applies only to source water samples, however once received by the laboratory, if sample processing does not begin on the same working day, samples must be refrigerated.

[7] If samples are analyzed after 30 hours, but within 48 hours of collection, the laboratory is to indicate in the analytical report that the data may be invalid because of excessive delay in sample processing. No samples received after 48 hours are to be accepted or analyzed for compliance with the regulations of the Department of Environmental Protection or the Department of Health.

[8] It is recommended that the preservative be added at the time of collection unless suspended solids activity is to be measured. It is also recommended that samples be filtered, if suspended or settleable solids are present, prior to adding preservative, at the time of collection. However, if the sample has to be shipped to a laboratory or storage area, acidification of the sample (in its original container) may be delayed for a period not to exceed 5 days. A minimum of 16 hours must elapse between acidification and analysis.

[9] If HCl is used to acidify samples, which are to be analyzed for gross alpha or gross beta activities, the acid salts must be converted to nitrate salts before transfer of the samples to planchets.

Table FS 1000-9
Containers, Preservation and Holding Times for Biosolids Samples and Protozoans

ANALYTE NAME	***CONTAINER***	***PRESERVATION***	***MAX HOLDING TIME***
Fecal Coliform	Plastic or Glass	Cool 4°C	24 hours
Salmonella	Plastic or Glass	< 10°C	24 hours
Enteric Viruses	Plastic or Glass	Up to 25°C	2 hours
Enteric Viruses	Plastic or Glass	2 to 10°C	48 hours
Specific Oxygen Uptake Rate	Plastic or Glass	None	As Soon As Possible
Helminth OVA	Plastic or Glass	< 4°C (Do not Freeze)	24 hours
Cryptosporidium/Giardia	Plastic or Glass	0 - 8°C (Do not Freeze)*	96 Hours
Total Solids	Plastic or Glass	≤6°C (Do not Freeze)	7 days
Metallics	Plastic or Glass	See Tables FS 1000-4, FS 1000-5 and FS 1000-6	
Other Inorganic Pollutants	Plastic or Glass	See Tables FS 1000-4, FS 1000-5 and FS 1000-6	

***Dechlorinate bulk samples when applicable**

Table FS 1000-10
Container Materials, Preservation, and Holding Times for Fish and Shellfish

Analyte	Matrix	Sample Container	Field (Transport to Lab)		Laboratory	
			Preservation	Maximum Shipping Time	Storage	Holding Time
	Whole Organism (Fish, shellfish, etc.	Foil-wrap each organism (or composite for shellfish) and transport in waterproof plastic bag	Cool in wet ice or: ——— Freeze on dry ice	24 hours ——— 48 hours		
Mercury	Tissue (fillets and edible portions, homogenates)	Plastic, borosilicate glass, quartz, PTFE			Freeze at <-20°C	28 days
Other metals	Tissue (fillets and edible portions, homogenates)	Plastic, borosilicate glass, quartz, PTFE			Freeze at <-20°C	6 months
Organics	Tissue (fillets and edible portions, homogenates)	Borosilicate glass, PTFE, quartz, aluminum foil			Freeze at <-20°C	1 year
Dioxin	Tissue (fillets and edible portions, homogenates)	Amber containers: Borosilicate glass, PTFE, quartz, aluminum foil			Freeze at <-20°C	30 days until extraction, 15 days after extraction
Lipids	Tissue (fillets and edible portions, homogenates)	Plastic, borosilicate glass, quartz, PTFE			Freeze at <-20°C	1 year

PTFE = Polytetrafluoroethylene (Teflon)

Table FS 1000-11
Holding Times for SPLP or TCLP Extraction, Sample Preparation and Determinative Analysis

Holding Time (Days)				
	From: Field Collection To: SPLP or TCLP Extraction	From: SPLP or TCLP Extraction To: Preparative Extraction	From: Preparative Extraction To: Determinative Analysis	Total Elapsed Time
Volatiles	14	NA	14	28
Semi-Volatiles	14	7	40	61
Mercury	28	NA	28	56
Metals, except Mercury	180	NA	180	360

NA – Not Applicable

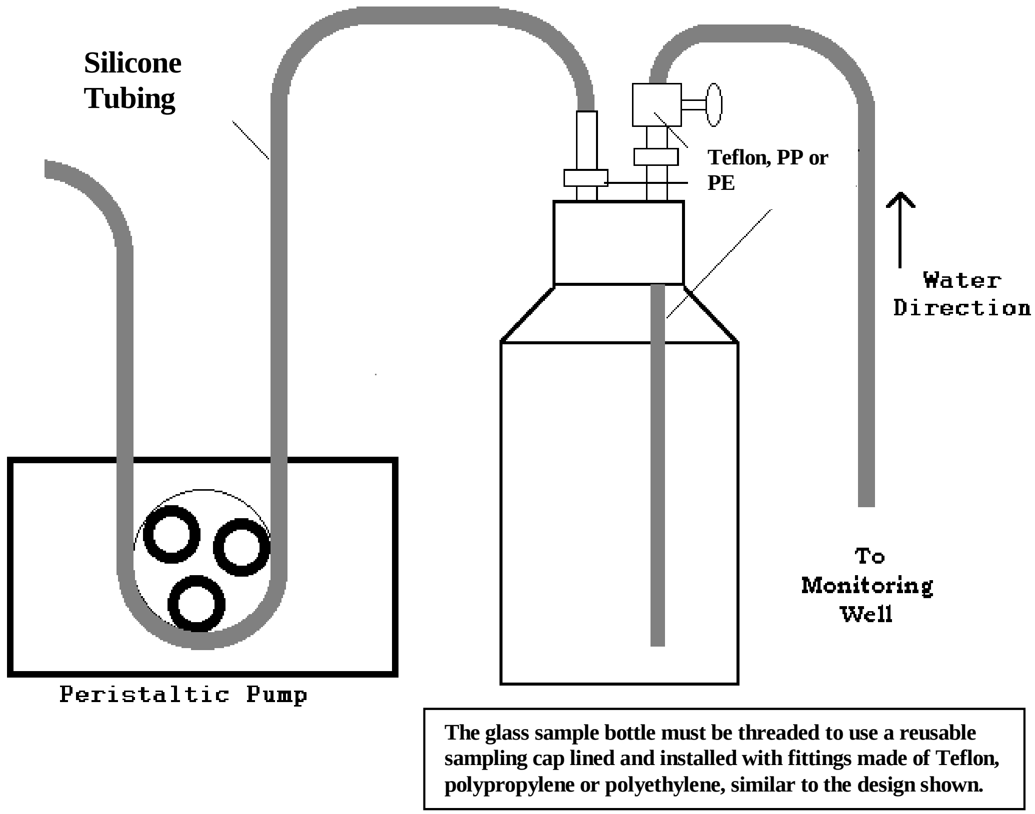
Table FS 1000-12
Preventive Maintenance Tasks

INSTRUMENT/ACTIVITY	FREQUENCY
REFRIGERATORS, INCUBATORS, OVENS	
Clean interior	Monthly
Check thermometer temperature against certified thermometer or equivalent	Annually
ANYTICAL BALANCES	
Clean pan and compartment	Daily[1]
Check with Class S weights	Monthly
Manufacturer cleaning and calibration	Annually
pH AND ION SELECTIVE ELECTRODES	
PROBE	
Check probe for cracks and proper levels of filling solution; check reference junction; clean electrode	Daily, Replace as necessary
Check response time	Daily[1]
METER	
Check batteries and electronics for loose connections and cracked leads	Daily[1], Replace as necessary
TURBIDIMETER	
Clean instrument housing	Monthly
Clean cells	Daily[1]
CONDUCTIVITY METER	
Check batteries and probe cables	Daily[1]
Replatinize Probe	Per manufacturer's recommendations
DISSOLVED OXYGEN METERS	
PROBE	
Check membrane for deterioration; check filling solution	Daily[1], Replace as necessary
METER	
Battery level and electronics checked	Daily[1], Replace as necessary
THERMOMETERS	
Check for cracks and gaps in the mercury	Daily[1], Replace as necessary
TEMPERATURE PROBE	
Check connections, cables	Daily[1]
Check against calibrated thermometer	Daily[1]
AUTOMATIC SAMPLE COLLECTION SYSTEMS (e.g., ISCO, Sigma)	
Check sampler operation (forward, reverse, automatic through three cycles of the purge-pump-purge cycle)	Daily[1]Prior to Sampling Event
Check purge-pump-purge cycle when sampler is installed	Daily[1]Prior to Sampling Event
Check the flow pacer that activates the sampler to assure proper operation	Daily[1]Prior to Sampling Event
Check desiccant	Daily[1], Replace as Necessary
Check batteries	Daily[1], Replace as Necessary
Check pumping rate against manufacturer's specifications	Daily[1], Replace as Necessary

[1]Daily is defined as prior to use or a 12-hour period if equipment is run continuously

Figure FS 1000-1

Organic Trap Configuration for Collecting Extractable Organics with a Peristaltic Pump



FS 2000. GENERAL AQUEOUS SAMPLING

See also the following Standard Operating Procedures:

- FA 1000 Administrative Procedures
- FC 1000 Cleaning/Decontamination Procedures
- FD 1000-9000 Documentation Procedures
- FM 1000 Field Planning and Mobilization
- FQ 1000 Field Quality Control Requirements

1. COMMON PROCEDURES

The following procedures are applicable to the collection of all water samples.

1.1. Refer to FS 1000 for procedures that are common to all types of sample collection including general preservation and thermal preservation procedures.

1.2. Grab Samples

1.2.1.1. This is an individual sample collected over a period of time, usually all in one motion, generally not exceeding 15 minutes. The 15-minute time limit applies to aqueous samples only. No time limit applies to the collection of solid samples (e.g., residuals).

1.2.1.2. Grab samples represent the conditions that exist at the moment the sample is collected and do not necessarily represent conditions at any other time. Grab sampling is the preferred method of sampling under the following conditions:

- A snapshot of the water quality at a particular instant in time is desired.
- The water or wastewater stream is not continuous (e.g., batch discharges or intermittent flow).
- The characteristics of the water or waste stream are known to be constant or nearly so.
- When conditions are relatively constant over the period of discharge. In lieu of complex sampling activities, a grab sample provides a simple and accurate method of establishing waste characteristics.
- The sample is to be analyzed for analytes whose characteristics are likely to change significantly with time (e.g., dissolved gases, microbiological tests, pH).
- The sample is to be collected for analytes such as Oil and Grease, bacteriological tests or other parameters listed in number 3 of this section where the compositing process could significantly affect the actual concentration.
- Data on maximum/minimum concentrations are desired for a continuous water or wastewater stream.
- When identifying and tracking slug loads and spills.

1.2.1.3. If required, measure the following parameters on grab samples or in-situ.

NOTE: If the permit specifies a composite sample for any of the parameters mentioned below, **FOLLOW THE PERMIT CONDITIONS**

Cyanide	Oil and Grease
Residual Chlorine	pH
Dissolved constituents in field-filtered samples (ortho-phosphorus, metals, etc.)	Specific Conductance
Dissolved Oxygen and other dissolved gases	Un-ionized Ammonia
Microbiological Parameters	Volatile Organic Compounds
TRPHs	Temperature
Total Phenols	

1.3. Composite Samples

1.3.1. A composite sample is a sample collected over time, formed either by continuous sampling or by mixing discrete samples. Composite samples reflect the average characteristics during the compositing period.

1.3.2. Composite samples are used when stipulated in a permit or when:

- The water or wastewater stream is continuous;
- Analytical capabilities are limited;
- Determining average pollutant concentration during the compositing period;
- Calculating mass/unit time loadings; or
- Associating average flow data to parameter concentrations

1.3.3. Composite samples may be collected individually at equal time intervals if the flow rate of the sample stream does not vary more than plus or minus ten percent of the average flow rate or they may be collected proportional to the flow rate. The permit or work plan will specify which composite sample type to use, either time composites or flow proportional composites. The compositing methods, all of which depend on either continuous or periodic sampling, are described in the following discussions.

1.3.3.1. Time Composite Sample: Time composite samples are based on a constant time interval between samples. A time composite sample can be collected manually or with an automatic sampler. This type of composite is composed of discrete sample aliquots collected in one container at constant time intervals. This method provides representative samples when the flow of the sampled wastewater stream is constant. This type of sample is similar to a sequential composite sample described in number 3.3 of this section.

1.3.3.2. Flow Proportional Composite Sample: Flow proportional samples can be collected automatically with an automatic sampler and a compatible pacing flow measuring device, semi-automatically with a flow chart and an automatic sampler capable of collecting discrete samples, or manually. There are two methods used to collect this type of sample:

- Method 1: Collect a constant sample volume per stream flow (e.g., a 200 mL sample collected for every 5,000 gallons of stream flow) at time intervals proportional to stream flow. This method provides representative samples of all waste streams when the flow is measured accurately.

- Method 2: Collect a sample by increasing the volume of each aliquot as the flow increases, while maintaining a constant time interval between the aliquots (e.g., hourly samples are taken with the sample volume being proportional to the flow at the time the sample is taken).

1.3.3.3. <u>Sequential Composite Sample</u>: Sequential composite samples are composed of discrete samples taken into individual containers at constant time intervals or constant discharge increments. For example, samples collected every 15 minutes are composited for each hour.

- The 24-hour composite is made up from the individual one-hour composites. Each of the 24 individual samples is manually flow-proportioned according to the flow recorded for the hour that the sample represents. Each flow-proportioned sample is then added to the composite samples. The actual compositing of the samples is done by hand and may be done in the field or the laboratory. In most cases, compositing in the field is preferable since only one sample container must be cooled, and then transported to, and handled, in the laboratory. A 24-hour composite is frequently used since an automatic sampler can easily collect the individual samples.

- A variation of the 24-hour composite is to collect a constant volume of sample taken at constant discharge increments, which are measured with a totalizer. For example, one aliquot is collected for every 10,000 gallons of flow

- Sequential sampling is useful to characterize the waste stream because you can determine the variability of the wastewater constituents over a daily period. For example, for pretreatment studies you can visually determine when high strength wastes are being discharged from a facility or when heavy solid loads are being discharged during a 24-hour cycle. You can measure the pH throughout the day. The value of this type of sampling must be weighed against the manpower constraints and sampling goals

1.3.3.4. <u>Continuous Composite Sample</u>: Collected continuously from the stream. The sample may be a constant volume that is similar to the time composite, or the volume may vary in proportion to the flow rate of the waste stream, in which case the sample is similar to the flow proportional composite.

1.3.3.5. <u>Areal Composite</u>: A sample composited from individual grab samples collected on an areal or cross-sectional basis. Areal composites must be made up of equal volumes of grab samples; each grab sample must be collected in an identical manner. Examples include residual samples from grid system points on a land application site, water samples collected at various depths at the same point or from quarter points in a stream, etc sample is similar to the flow proportional composite.

1.4. <u>Collection Techniques</u>

1.4.1. When filling a sample container that already contains premeasured preservative, slowly pour the sample down the side of the container so that the preservative does not splatter. If the preservative is concentrated acid, and the sample water is added too quickly, the reaction between the water and the acid can generate enough heat to burn unprotected skin or could splatter and cause acid burning.

1.4.2. Collect grab samples (single, discrete samples) unless directed by permit, program, or approved sampling plan or work plan to collect composite samples.

1.4.3. Except for volatile organic compounds and sulfide, leave ample headspace in the sample bottle to allow for expansion, effervescence and proper mixing at the laboratory.

1.5. Collecting Filtered/Dissolved Samples

1.5.1. Certain studies or projects require collection of dissolved (i.e., filtered) samples. Identify all analytes in samples that are filtered as “dissolved” or “filtered” in field notes or laboratory transmittal forms and on final reports.

1.5.2. Collect both filtered and unfiltered samples from the same water in a collection device (e.g., bailer, intermediate container) or consecutively if sampling from a pump.

1.5.3. Collect dissolved metals in groundwater according to the procedures discussed in FS 2225. **Do not** collect filtered samples for metals from groundwater sources unless:

1.5.3.1. The DEP has required or approved the protocol and the DEP program allows the use of the procedure; or

1.5.3.2. The organization is documenting that a filtered groundwater sample is as or more representative of the groundwater quality. In this case, collect **both** unfiltered and filtered samples for analysis. Submit the results of both samples the DEP for review.

1.5.4. Filtration, when performed, must begin within 15 minutes of sample collection.

1.5.5. Collect dissolved groundwater samples for metals with a one-piece molded construction 1 μm filter unless otherwise specified by a DEP program. Use a 0.45 μm filter when filtering all other constituents **including** metals in surface water.

1.5.6. The filter must be compatible with the analyte to be filtered (e.g., zero carbon content for carbon analysis; non-protein binding filters for nitrogen).

1.5.7. Equipment blanks, when collected, must be processed through the filtration apparatus and analyzed for the analytes of interest.

1.5.8. Filters and filtration equipment are intermediate devices and therefore must be adequately rinsed per FS 2110 section 1.1.2.1.

THE FOLLOWING ARE SPECIAL CONSIDERATIONS FOR VARIOUS ANALYTE GROUPS:

FS 2001. *pH-Preserved Samples*

1. SAMPLE CONTAINERS

1.1. Use properly cleaned sample containers (see FC 1300).

1.2. Inspect all containers for visual defects or contamination. Discard if defects are present or containers do not appear clean.

2. SAMPLE COLLECTION PROCEDURES

2.1. Perform any filtration **before** the sample is poured into the container and **before** the sample is preserved.

2.2. Remove the cap from the sample container, and carefully pour the sample into the container without allowing sampling equipment or hands to touch the rim of the sample container.

2.3. If the preservative is added after the sample is collected, (the container is not prepreserved), do not fill the container to the rim.

3. PRESERVATION

3.1. Preserve the sample within 15 minutes of sample collection or filtration (if applicable) unless collected as a composite sample (see FS 1006, section 1.3) or for analysis of lead and copper for drinking water compliance (see FS 2310, section 2).

3.2. Preserve the sample with the chemical specified by the method or preservation tables (Tables FS 1000-4 to FS 1000-10).

3.2.1. The chemical reagents must be pure enough so that the reagent does not contribute contamination or interferences to the analytes of interest.

3.3. Preserve the sample by adding an accurately measured amount of preservative to the container. Premeasured vials of the preservative, or a graduated container or pipet, may be used.

3.3.1. Tightly cap the sample container and gently tip the container two to three times to distribute the chemical.

3.4. The pH of the preserved sample must meet the pH criterion of the applicable preservation tables (see Tables FS 1000-4 to FS 1000-10). **Do not over preserve the sample.**

3.4.1. Pour an aliquot of the preserved sample into a disposable container (e.g., sampling cup) or onto a piece of **narrow** range pH paper to determine if the pH meets the required level. **Do not put the pH paper directly into the sample container.**

3.4.2. If the pH does not meet the required level, add additional measured amounts of preservative and test with narrow range pH paper (see section 3.4.1 above) until the pH meets the pH requirement.

3.4.3. Record the total amount of preservative that was added to the sample. This documentation is necessary for the next site visit, since additional acid may be needed to adequately preserve the sample on subsequent visits.

3.5. Cooling to less than 6°C with wet ice (see FS 1006, section 5) may be required.

3.6. Protect from direct sunlight.

3.7. Preserve at least one of the equipment blanks with the **greatest** amount of preservative that was required in the sample set and note the amount in field documentation.

3.8. After the sample has been preserved, screw the cap on tightly.

4. <u>Verifying pH-Preserved Samples:</u> Verify the pH of all pH-preserved samples (except volatile organics) in the field (see FS 2001, section 3.4). If samples are routinely collected from the same sample location, a pH check is not required each time samples are collected.

4.1. If the frequency of sample collection at a specified location is once per month or greater (e.g., weekly or daily), check the pH of **at least one** sample per parameter group according to the following schedule:

4.1.1. Weekly sampling: 1 pH check per month

4.1.2. Daily sampling: 1 pH check per week

4.2. If the frequency of sample collection at a specified location is once per month, check the pH of at least one sample per parameter group (except volatile organics) quarterly.

4.2.1. If site conditions vary from sampling event to sampling event, perform pH checks at increased intervals.

4.2.2. For all other sample collection frequencies, pH checks may be reduced as follows:

4.2.2.1. During the first sampling event at a particular site, check **all** samples (except volatile organics) that are pH-adjusted, and

4.2.2.2. During subsequent visits to a particular site, check **at least one** sample per parameter group that must be pH-adjusted.

5. DOCUMENTATION

5.1. Complete the sample container label and stick firmly on the container.

5.2. Complete the field notes.

5.3. Make notes on the transmittal form and in field records about any relevant observations or problems such as entrained sediment or preservation problems.

FS 2002. *Metals*

1. SAMPLE CONTAINERS

1.1. Use properly cleaned containers (see FC 1300).

1.2. Inspect the containers and caps for visual defects or contamination. Do not use containers if defects are present or if they do not appear clean.

2. SAMPLE COLLECTION PROCEDURES

2.1. Perform any filtration **before** the sample is poured into the container and **before** the sample is preserved.

2.2. Remove the cap from the sample container and carefully pour the sample into the container without allowing sampling equipment or hands to touch the rim of the sample container.

3. PRESERVATION - Follow preservation procedures outlined in FS 2001 above.

3.1. Requirements for specific metals:

3.1.1. For boron or cold-vapor atomic absorption Mercury with a grade of nitric acid (HNO_3) that is suitable for use for metals analysis. Use concentrated HNO_3 or 1:1 HNO_3.to lower the pH of less than 2 S.U., but greater than 1.62 S.U.

3.1.2. For Chromium VI add sufficient ammonium sulfate buffer solution specified per Table FS 1000-4 to the sample to raise the pH of the sample to a pH of 9.3 – 9.7 and place in ice (see FS 2002).

3.1.3. <u>Trace Level Mercury</u>

3.1.3.1. Collect samples for trace level mercury (<100 ug/L) in tightly-capped fluoropolymer or glass bottles.

3.1.3.2. If the samples cannot be received by the laboratory within 48 hours of sample collection, preserve the sample with BrCl or HCl solution.

3.1.3.3. For dissolved trace level mercury, samples must be filtered through a 0.45 µm filter within 24 hours of sample collection. If the samples cannot be transported to the laboratory within 24 hours, follow the procedures in FS 8200 for field filtration.

3.1.4. Samples collected for lead and copper for drinking water compliance and metals other than those listed above do not require immediate acid preservation.

3.1.4.1. When samples are not acidified with acid, the transmittal form to the laboratory must:

- Clearly state that the samples are unpreserved; and
- Request that the laboratory preserve the samples.

3.1.4.2. If samples are acidified, use concentrated HNO_3 or 1:1 HNO_3.to lower the pH of less than 2 S.U., but greater than 1.62 S.U.

3.2. After the sample has been preserved, screw the cap on tightly.

4. DOCUMENTATION

4.1. Complete the sample container label and stick firmly on the container.

4.2. Complete the field notes.

4.3. Make notes on the transmittal form and in field records about any relevant observations or problems such as entrained sediment.

4.4. On the transmittal form, clearly identify samples that must be acidified by the laboratory (FS 2002, 3.1.3 or 3.1.4 above).

FS 2003. *Extractable Organics*

1. SAMPLE CONTAINERS

1.1. Most samples are collected in glass containers with Teflon-lined caps. Note: Teflon containers are also acceptable. There are some exceptions such as collecting samples in amber glass (e.g., nitroamines, nitroaromatics, etc.). If in doubt, verify the proper container type in Tables FS 1000-4 through FS 1000-10.

1.2. Inspect glass bottles to assure that there are no visual glass or liner defects. If defects are present and/or the sample containers do not appear clean, the bottles must be discarded.

1.3. Collect composite samples from automatic sample collection devices in refrigerated glass or Teflon containers through Teflon, polyethylene or polypropylene tubing.

2. SAMPLE COLLECTION PROCEDURES

2.1. Remove the cap from the sample container without touching the interior Teflon liner.

2.2. Carefully pour the sample into the container without allowing sampling equipment or hands to touch the rim of the sample container.

2.3. Fill bottle with sample to almost full capacity.

3. PRESERVATION

3.1. In general, these types of samples must be preserved by cooling to 4°C.

3.1.1. Some analyte groups require a chemical preservation. See Tables FS 1000-4 through FS 1000-10 for any additional preservation.

3.1.2. If the samples for pesticides cannot be extracted within 72 hours of collection, the sample pH must be in the range of 5 to 9. If needed, adjust sample to the specified pH range with sodium hydroxide or sulfuric acid.

3.1.3. Add sodium thiosulfate if residual chlorine is present.

3.2. Place samples in **wet** ice within 15 minutes of sample collection (see FS 1006, section 5).

4. DOCUMENTATION

4.1. Complete the sample container label and stick firmly on the container.

4.2. Document when samples were placed in wet ice immediately (see FS 1006, section 5).

4.3. Complete the field notes.

4.4. Make notes on the lab transmittal form and the field records about any sample that appears highly contaminated or exhibits other abnormal characteristics (i.e., foaming, odor, etc.).

FS 2004. *Volatile Organics*

1. SAMPLE CONTAINERS

1.1. Use a screw cap glass sample vial that is sealed with a Teflon-coated septum.

1.2. Collect **at least two** vials of each sample. Some laboratories may require three or more vials, therefore verify the laboratory's policy on the number of vials they require unless the laboratory provides the sampling kit.

1.3. Inspect the vials for glass or septum defects (e.g., rim must not have nicks or visible depressions and the septum must not be deformed). Do not use containers if defects are present or if they do not appear clean.

2. SAMPLE COLLECTION PROCEDURES

2.1. Special precautions for petroleum sources:

2.1.1. If possible, transport and store fuels in a separate vehicle from sampling equipment, empty vials and collected samples. If these items must be transported in the same vehicle as fuel, store the fuels as far away from the vials as possible.

2.1.2. Place all fuel or exhaust sources downwind of the sampling location.

2.1.3. Position all petroleum-fueled engines (including the vehicle) downwind of the sampling operations.

2.2. Do not allow the sampling equipment or hands to touch the rim of the sample container.

2.3. Do not remove septum caps from VOC vials until just prior to filling. Cap vials immediately after filling with sample.

2.4. **DO NOT PRERINSE VOC VIALS.**

2.5. Do not aerate the sample during sample collection. If collecting from a spigot or pump, reduce the flow rate to less than 100 mL/min.

2.6. If preservation is required, proceed to section 3 below unless the laboratory supplied vials with premeasured quantities of acid, and the sample does not need to be dechlorinated (see 3.2 below).

2.6.1. If no preservation is required or if the vials are prepreserved (see 2.5 above), slowly and carefully allow the sample to flow down the **side** of the vial to minimize turbulence. Fill the vial until the surface tension holds the water in a "convex meniscus".

2.6.2. If a vial overflows during the filling process, document the problem and notify the laboratory that the vial may not contain sufficient acid.

2.6.3. If using a bailer, the bailer must be equipped with a controlled flow bottom assembly.

3. PRESERVATION

3.1. Preserve the sample **during** the sample collection process.

3.2. Dechlorination: Some treated water samples (drinking water and treated wastewater) may contain residual chlorine that must be removed with a declorination agent such as sodium thiosulfate or ascorbic acid. This process must occur **before** any additional preservatives (i.e., acid) are added. The dechlorination agent must be **in the vial** before the sample is added.

3.2.1. Laboratories may supply vials with premeasured quantities of declorination agent. If acid preservation **is not required**, fill the vials (see section 2.5.1 above) and proceed to section 4 below.

3.2.2. For chlorinated drinking water samples, add 3 mg sodium thiosulfate per 40 mL vial.

3.2.3. If the chlorine level is unknown, the concentration must be measured (see FT 2000). For sources other than drinking water (e.g., chlorinated effluent), 10 mg sodium thiosulfate per 40 mL vial will remove up to 5 ppm Cl_2.

3.3. Acid Preservation

3.3.1. Chlorinated Samples

3.3.1.1. If acid preservation is required, carefully fill the vial with sample, but not to a convex meniscus as described in section 2.5.1 above.

3.3.1.2. Add four drops of concentrated HCl (more acid may be needed if the sample is known to contain high levels of bicarbonate or is otherwise buffered).

3.3.1.3. Add additional sample to create a convex meniscus.

NOTE: If the sample reacts with the acid by generating gas, do not submit preserved samples for analysis. Instead, collect unpreserved samples (seven-day holding time must be met).

3.3.2. Unchlorinated Samples

3.3.2.1. The laboratory may supply vials with premeasured quantities of acid. In this case, proceed to section 2.5.1 above. If a vial overflows during the filling process, document the problem and notify the laboratory that the vial may not contain sufficient acid.

3.3.2.2. If the samples are preserved in the field, follow the procedure in section 3.3 above.

4. CAPPING THE VIAL

4.1. Fill the vial so that the sample surface is above the container rim (convex meniscus).

4.1.1. **Do not pour** sample into cap.

4.1.2. Fill vial from the original source (tubing, spigot, etc.) **Do not fill vial from sample collected in the cap**.

4.2. **Immediately** cap the vial with the Teflon seal contacting the sample. Some sample may overflow while tightening the cap.

4.3. If acid has been added to the sample, tip the vial gently two or three times to distribute the preservative.

4.4. Turn the vial over and tap it to check for the presence of bubbles.

4.4.1. If bubbles are present, and the total volume of the bubbles is less than 5 mm in diameter, the sample may be submitted.

4.4.2. If the total volume of the bubbles is greater than 5 mm in diameter, discard the vial and fill a new one.

4.4.3. **Do not open a vial to add additional sample.**

5. SAMPLE PACKING

5.1. Label each vial with an appropriate field ID number and preservation (e.g., preserved with acid, sodium thiosulfate/acid, etc.).

5.2. Wrap each vial in a protective material (e.g., bubble wrap).

5.3. Place the set of vials in a small, sealable, untreated plastic bag unless the laboratory supplies an alternate method of packing.

5.4. Place samples in **wet** ice within 15 minutes of sample collection (see FS 1006, section 5).

5.5. Protect samples from environmental contamination during storage and transport to the laboratory.

5.6. As an added measure, DEP recommends wrapping the set of replicate samples in bubble wrap and sealing them in a container. This procedure will add further protection from potential contamination.

6. DOCUMENTATION

6.1. Label all the vials.

6.2. Complete field records.

6.3. Make note in the field records of any samples that appear highly contaminated or appear to effervesce when acid is added.

FS 2005. *Bacteriological Sampling*

1. SAMPLE CONTAINERS

1.1. Collect the samples in properly sterilized containers.

1.1.1. Presterilized Whirl-pak bags (or equivalent) are generally used.

1.1.2. If Whirl-pak bags are not used, the sample container must have a volume of at least 125 mL.

1.1.3. If using bottles, the caps must be sterilized. If the caps are lined, there must be documentation to show that the liner does not produce toxic compounds when sterilized.

1.1.4. Bottles and caps must be sterilized according to procedures in FC 1320 or purchased presterilized from a commercial vendor.

2. SAMPLE COLLECTION PROCEDURES

2.1. Unless a composite is specified by permit, all samples must be grab samples.

2.2. Do not open the container once it has been sealed.

2.3. Do not rinse sample container before collecting the sample.

2.4. Use aseptic techniques to collect the sample:

2.4.1. If an intermediate device is used, thoroughly rinse with sample water. To ensure proper rinsing, DEP recommends that microbiological samples be the last sample collected with the sampling device.

2.4.2. Do not put fingers into the mouth of the container or on the interior of the cap.

2.4.3. Do not disinfect the sample equipment or sampling port.

2.5. Rinse the sampling equipment with sample water before collecting the sample. Therefore, collect microbiological samples at the end of a sampling sequence.

2.6. <u>Wells with In-Place Plumbing, Spigots and/or Faucets</u>

2.6.1. Do not disinfect the spigot with bleach, alcohol or heat. Turn on spigot and flush at maximum velocity (see FS 2310).

2.6.2. After flushing, reduce the water flow to approximately 500 mL/min and allow the water to flow for a few minutes before collecting samples. If other samples (metals, nutrients, etc.) are to be collected, collect these samples first.

2.6.3. **Do not stop the flow before or during the filling process.**

2.7. <u>Direct Grab Sample Collection</u>

2.7.1. Hold a rigid container near the base and plunge neck downward, below the surface. Turn container until the neck points slightly upward with the mouth directed toward the current. Fill to within about 1/2 inch of the top and cap immediately.

2.7.2. <u>Whirl-pak bags (or equivalent)</u>

- Open the bag by zipping off the top and pulling the white tabs to open the bag. Hold the bag behind the wire ties, and plunge neck downward and up in one sweeping arc; or
- Zip off the top of the bag. Hold bag so that the mouth and wire ties are in front of the hands and fingers. Immerse the bag, and open the bag into the current.
- The above procedures may also be accomplished by attaching the bag to a pole.

2.7.2.1. Bring the bag to the surface, and press out excess water.

2.7.2.2. Seal the bag by folding the open ends at least three times and securely twisting the wire ties.

2.8. Intermediate Device Collection

2.8.1. When using an intermediate sampling device (bailer, DO dunker, niskin bottle, etc.), obtain sufficient sample in the sample collection device to completely fill the sample container. Begin pouring sample out of the device BEFORE collecting into the container. Continue to pour sample out of the device, place container under flowing stream, and fill. **Do not stop the flow before or during the filling process.**

3. PRESERVATION

3.1. Preserve samples according to Tables FS 1000-4 through FS 1000-10.

3.2. Place all samples in wet ice immediately after sample collection (see FS 1006, section 5).

3.3. When the sample contains residual chlorine, add a dechlorinating agent such as sodium thiosulfate to the sample container.

3.3.1. The final concentration of sodium thiosulfate must be approximately 100 milligrams per liter (mg/L) in the sample (add 0.1 mL of a 10% solution of thiosulfate to a 125 mL sample).

3.3.2. Some vendors or laboratories provide sterile containers with premeasured amounts of dechlorinating agent. Determine if the source of the field containers already contain a dechlorinating agent.

3.3.3. **Do not use containers with dechlorinating chemicals** when collecting samples from sources that are known to be free from residual chlorine.

4. HOLDING TIME

4.1. The holding time for microbiological samples is very short. Let the laboratory know the approximate time that samples will be collected and when they are expected to be delivered to the laboratory.

4.2. The holding time begins at the time (hours and minutes) the sample is collected and ends at the time that the sample is placed on the applicable growth media.

4.3. Consult Tables FS 1000-4, -6, -8, and -9 for holding times.

5. DOCUMENTATION

5.1. Label each sample container with an appropriate field ID number.

5.2. Place samples in **wet** ice within 15 minutes of sample collection (see FS 1006, section 5).

5.3. Complete field records.

5.4. Make note in the field records of any unusual sample appearances or sampling conditions.

FS 2006. *Oil and Grease (O&G) and Total Recoverable Petroleum Hydrocarbons (TRPHs)*

1. SAMPLE CONTAINERS

1.1. Collect samples for O&G and TRPHs in 1-liter wide mouth amber glass bottles.

1.2. The cap must have a Teflon liner.

1.3. Visually inspect glass bottles and caps for defects. Do not use container if defects are present or if they do not appear clean.

2. SELECTION OF SAMPLING POINTS

2.1. Oil and grease may be present in wastewater as a surface film, an emulsion, a solution, or as a combination of these forms. Since it is very difficult to collect a representative ambient sample for oil and grease analysis, the sampler must carefully evaluate the location of the sampling point.

2.1.1. Select a point of greatest mixing.

2.1.2. For compliance samples at a facility, collect samples from a point that best represents oil and grease concentrations.

3. SAMPLE COLLECTION PROCEDURES

3.1. All samples must be grab samples.

3.1.1. If composite data are required, collect individual grab samples over the specified time period.

3.1.2. Submit all samples for analysis.

3.1.3. Average the concentrations of the results to determine the average concentration over time.

3.2. Do not collect the sample by skimming the surface.

3.3. Collect a discrete sample that will be used for analysis. Do not use this sample for any other test.

3.4. Remove the cap from the glass bottle without touching the interior of the container or lid.

3.5. Do not rinse the sampling device or the sample container with sample water.

3.6. Collect the sample directly into the container.

3.6.1. If intermediate sampling equipment is needed, do not allow the sampling equipment to touch the rim of the sample container.

3.6.2. Do not use automatic samplers to collect these types of samples.

3.6.3. Fill the bottle with the sample water to almost full capacity.

3.6.4. Add preservatives (see section 4 below).

3.6.5. Quickly cap the container and tighten securely.

4. PRESERVATION

4.1. Preserve the sample within 15 minutes of sample collection.

4.2. The pH of the acidified sample must be less than 2. **Do not over acidify the sample.**

4.3. Preserve the sample by adding an accurately measured amount of sulfuric or hydrochloric acid to the container. Premeasured vials of acid, or a graduated container or pipet, may be used.

4.3.1. Tightly cap the sample container and shake to distribute the acid.

4.3.2. Pour an aliquot of the acidified sample into a disposable container (e.g., sampling cup) or onto a piece of **narrow** range pH paper to determine if the pH is less than 2. **Do not put the pH paper directly into the sample container.**

4.3.3. If the pH is greater than 2, add additional measured amounts of acid and test with narrow range pH paper (see section 4.3.2 above) until the pH has been reduced to below 2 pH units.

4.3.4. Record the total amount of acid that was added to the sample.

4.4. Acidify at least one of the equipment blanks with the **greatest** amount of acid that was required in the sample set and note the amount in field documentation.

4.5. After the sample has been preserved, screw the cap on tightly.

4.6. Immediately place the sample in **wet** ice after preserving with acid (see FS 1006, section 5).

5. DOCUMENTATION

5.1. Label each vial with an appropriate field ID number.

5.2. Protect glass container from breakage ("bubble wrap" is recommended).

5.3. Complete field records.

5.4. Make notes on the transmittal form and in field records about any relevant observations or problems such as entrained sediment.

FS 2007. *Radiological Sampling (Excludes Radon)*

1. SAMPLE CONTAINERS

1.1. Use polyethylene, polyvinyl chloride (PVC), or Teflon containers.

1.2. Visually inspect the containers and caps for defects. If defects are present and/or sample containers do not appear to be clean, do not use the containers.

2. SAMPLE COLLECTION PROCEDURES

2.1. On unknown sites, survey the area with a beta-gamma survey instrument, such as a Geiger-Müller meter.

2.1.1. If radiation levels are above instrument background, consult a radiation safety specialist to determine appropriate safety procedures.

2.2. Remove the cap from the sample container and carefully pour the sample into the container without allowing sampling equipment or hands to touch the rim of the sample container.

3. PRESERVATION

3.1. Preserve the sample with a suitable grade of nitric acid (HNO_3).

3.2. Preserve the sample within 15 minutes of sample collection.

3.3. The pH of the acidified sample must be less than 2. **Do not over acidify the sample.**

3.4. If the preservative is added after the sample is collected (the container is not prepreserved), do not fill the container to the rim.

3.5. Preserve the sample by adding an accurately measured volume of concentrated HNO_3 or 1:1 HNO_3 to the container. Premeasured vials of acid, or a graduated container or pipet, may be used.

3.5.1. Tightly cap the sample container and shake to distribute the acid.

3.5.2. Pour an aliquot of the acidified sample into a disposable container (e.g., sampling cup) or onto a piece of **narrow** range pH paper to determine if the pH is less than 2. **Do not put the pH paper directly into the sample container.**

3.5.3. If the pH is greater than 2, add additional measured amounts of acid and test with narrow range pH paper (see section 3.5.2 above) until the pH has been reduced to just below 2 pH units.

3.5.4. Record the total amount of acid that was added to the sample.

3.5.5. Cooling to 4°C is not required.

3.6. Acidify at least one of the equipment blanks with the **greatest** amount of acid that was required in the sample set and note the amount in field documentation.

3.7. After the sample has been preserved, screw the cap on tightly.

4. DOCUMENTATION

4.1. Complete the sample container label and stick firmly on the container.

4.2. Complete the field notes.

4.3. Make notes on the transmittal form and in field records about any relevant observations or problems such as entrained sediment.

FS 2008. *Radon Sampling*

Radon is a gas and is easily removed from water sources. Therefore, follow the same precautions and care used to collect volatile organic samples. Minimize contact with air during sample collection. Other sample collection techniques may be appropriate, depending on the analytical method or as specified in the project data quality objectives.

1. SAMPLE CONTAINERS

1.1. Use glass sample vials containing a premeasured portion of the scintillation "cocktail."

1.2. Visually inspect the containers and caps for defects. If defects are present and/or sample containers do not appear to be clean, do not use the containers.

1.3. Collect at least two samples.

2. PRESERVATION: The scintillation cocktail is the only required preservative.

3. SAMPLE COLLECTION PROCEDURES Obtain specific sample collection instructions from the laboratory that will analyze the samples. These instructions must include proper handling as well as sample size and packing instructions. The following are general instructions for collecting the samples:

3.1. Carefully fill a syringe (usually 10 mL) with sample water so that air bubbles are not pulled in with the sample before, during or after filling.

3.2. Place the tip of the syringe BELOW the scintillation cocktail and slowly dispense the sample BENEATH the cocktail surface.

3.3. Replace the lid and cap tightly.

3.4. Generally, the vial is used in the laboratory analytical instrument and labels or ID numbers on the sides of the containers may interfere with the analysis. Check with the laboratory for proper placement of labels or field ID numbers.

3.5. Ship in an upright position in the shipping containers that have been provided by the laboratory. If none are provided, protect vials from breakage ("bubble wrap" is recommended), segregate replicate samples in separate plastic bags, and ship to the laboratory in an upright position.

4. DOCUMENTATION

4.1. Complete the field notes.

4.2. Make notes on the transmittal form and in field records about any relevant observations or problems such as entrained sediment.

FS 2009. *Cyanide Sampling*

Cyanide is a very reactive and unstable species and is highly toxic. Samples suspected of containing cyanide must be handled very carefully.

1. SAMPLE CONTAINERS

1.1. Use polyethylene or glass sample containers.

1.2. Use properly cleaned containers (see FC 1300).

1.3. Visually inspect the containers and caps for defects. If defects are present and/or sample containers do not appear to be clean, do not use the containers.

2. SAMPLE COLLECTION PROCEDURES

2.1. Remove the cap from the sample container, and carefully pour the sample into the container without allowing sampling equipment or hands to touch the rim of the sample container.

3. PRESERVATION

3.1. Many different analytes interfere with the cyanide analysis (e.g., sulfides). If any interferences are known to be present, pretreat the sample for interferences by following the applicable footnotes in Table FS 1000-4.

3.2. Preserve the sample within 15 minutes of sample collection.

3.3. Preserve samples with sodium hydroxide to a pH greater than 12.

3.4. Preserve the sample by adding an accurately measured amount of a sodium hydroxide solution or sodium hydroxide pellets to the container. Use a graduated container or pipet to add the solution.

3.4.1. Tightly cap the sample container and shake to distribute the preservative.

3.4.2. Pour an aliquot of the preserved sample into a disposable container (e.g., sampling cup) or onto a piece of **narrow** range pH paper to determine if the pH is greater than 12. **Do not put the pH paper directly into the sample container.**

3.4.3. If the pH is less than 12, add additional measured amounts of the preservative and test with narrow range pH paper (see section 3.4.2 above) until the pH has been raised to above 12 pH units.

3.4.4. Record the total amount of preservative that was added to the sample.

3.5. After the sample has been preserved, screw the cap on tightly.

3.6. Immediately put the sample in **wet** ice (see FS 1006, section 5).

3.7. Preserve at least one of the equipment blanks with all the reagents and the **greatest** amount of sodium hydroxide that was required in the sample set and note the amount in field documentation.

4. DOCUMENTATION

4.1. Complete the sample container label and stick firmly on the container.

4.2. Complete the field notes.

4.3. Make notes on the transmittal form and in field records about any relevant observations or problems such as entrained sediment.

4.4. Ensure that all preservation measures are part of the field notes.

FS 2010 *Sulfide Sampling*

1. Analyze samples within 15 minutes of collection, or the preserve the sample within 15 minutes for later analysis. If preservation is required add the zinc acetate and sodium hydroxide to the container **before** filling with sample.

2. Avoid aerating the sample during collection. Slowly pour the sample slowly and carefully allow the sample to flow down the **side** of the container to minimize turbulence.

3. Check the pH (if necessary) before completing the filling process.

4. Complete the filling process. **Do not leave a head space.**

FS 2200. Groundwater Sampling

1. INTRODUCTION AND SCOPE

1.1 Use these Standard Operating Procedures to collect groundwater samples. They are designed to ensure that the collected samples will be representative of water in the aquifer or target formation and that the samples have not been altered or contaminated by the sampling and handling procedures. These procedures apply to permanently and temporarily installed monitoring wells, wells constructed using “direct-push” techniques, wells with installed plumbing, remedial groundwater treatment systems and excavations where groundwater is present. Use of alternative, DEP-approved and properly documented procedures (e.g., Corporate SOP, ASTM Standards, alternative equipment, etc.) is acceptable if they meet the intent (e.g., sample representativeness and integrity) of this standard (see FA 1000).

1.2 The topics in this SOP include equipment and supply selection, equipment construction materials, and purging and sampling techniques.

1.3 Use the following DEP SOPs in conjunction with FS 2200:

- FA 1000 Regulatory Scope and Administrative Procedures for Use of DEP SOPs
- FC 1000 Cleaning/Decontamination Procedures
- FD 1000 Documentation Procedures
- FQ 1000 Field Quality Control Requirements
- FS 1000 General Sampling Procedures
- FS 2000 General Aqueous Sampling
- FT 1000 Field Testing and Measurement
- FT 1100 Field pH
- FT 1200 Field Specific Conductance
- FT 1400 Field Temperature
- FT 1500 Field Dissolved Oxygen
- FT 1600 Field Turbidity

2. Groundwater samples may be collected from a number of different configurations. Each configuration is associated with a unique set of sampling equipment requirements and techniques:

3. <u>Wells without Plumbing</u>: These wells require that equipment be brought to the well to purge and sample unless dedicated equipment is placed in the well.

4. <u>Wells with In-Place Plumbing</u>: Wells with in-place plumbing do not require that equipment be brought to the well to purge and sample. In-place plumbing is generally considered permanent equipment routinely used for purposes other than purging and sampling, such as for water supply. They are generally found at wellfields, industrial facilities, and private residences. See FS 2300 for procedures to sample potable water wells. Air Strippers or Remedial Systems: These types of systems are installed as remediation devices. Sample these wells like drinking water wells (see FS 2300).

FS 2201 *Equipment and Supplies*

Use groundwater purging and sampling equipment constructed of only non-reactive, non-leachable materials that are compatible with the environment and the selected analytes. In selecting groundwater purging and sampling equipment, give consideration to the depth of the well, the depth to groundwater, the volume of water to be evacuated, the sampling and purging technique, and the analytes of interest. Refer to Tables FS 1000-1, FS 1000-2, FS 1000-3 and FS 2200-1 for selection of appropriate equipment.

Additional supplies such as reagents, preservatives, and field measurement equipment are often necessary.

1. FLOW CONTAINER: DEP recommends using a flow-through cell or container when collecting measurements for purging stabilization. The design must ensure that fresh formation water continuously contacts the measuring devices and does not aerate the sample or otherwise affect the groundwater properties.

2. PUMPS: All pumps or pump tubing must be lowered and retrieved from the well slowly and carefully to minimize disturbance to the formation water. This is especially critical at the air/water interface. Avoid the resuspension of sediment particles (turbidity) at the bottom of the well or adhered to the well casing during positioning of the pump or tubing.

2.1 Above-Ground Pumps

2.1.1 Variable Speed Peristaltic Pump: Use a variable speed peristaltic pump to purge groundwater from wells when the static water level in the well is no greater than 20-25 feet below land surface (BLS). If the water levels are deeper than 18-20 feet BLS, the pumping velocity will decrease.

2.1.1.1 A variable speed peristaltic pump can be used for normal purging and sampling (see FS 2213 and FS 2221), sampling low permeability aquifers or formations (see FS 2222) and collecting filtered groundwater samples (see FS 2225, section 1).

2.1.1.2 Most analyte groups can be sampled with a peristaltic pump if the tubing and pump configurations are appropriate. See Table FS 1000-3 for proper tubing selection and pump configurations.

2.1.2 Variable Speed Centrifugal Pump: A variable speed centrifugal pump can be used to purge groundwater from 2-inch and larger internal diameter wells. Do not use this type of pump to collect groundwater samples.

2.1.2.1 When purging is complete, do not allow the water that remains in the tubing to fall back into the well. Install a check valve at the end of the purge tubing, and withdraw the tubing slowly from the well while the pump is still running.

2.1.2.2 See Table FS 1000-3 for proper tubing selection and allowable analyte groups.

2.2 Submersible Pumps

2.2.1 Variable Speed Electric Submersible Pump: A variable speed submersible pump can be used to purge and sample groundwater from 2-inch and larger internal diameter wells.

2.2.1.1 A variable speed submersible pump can be used for normal purging and sampling (see FS 2213 and FS 2221), sampling low permeability aquifers or formations (see FS 2222) and collecting filtered groundwater samples (see FS 2225, section 1).

2.2.1.2 Make sure that the pump housing, fittings, check valves and associated hardware are constructed of stainless steel. Make sure that any other materials are compatible with the analytes of interest. See Table FS 1000-3 for restrictions.

2.2.1.3 Install a check valve at the output side of the pump to prevent backflow.

2.2.1.4 If purging and sampling for organics:

- The entire length of the delivery tube must be Teflon, Polyethylene or Polypropylene (PP) tubing.
- The electrical cord must be sealed in Teflon, Polyethylene or PP and any cabling must be sealed in Teflon, Polyethylene or PP, or be constructed of stainless steel.
- All interior components that contact the sample water (impeller, seals, gaskets, etc.) must be constructed of stainless steel or Teflon.

2.2.2 <u>Variable Speed Bladder Pump</u>: A variable speed positive displacement bladder pump (no-gas contact) can be used to purge and sample groundwater from 3/4-inch and larger internal diameter wells.

2.2.2.1 A variable speed bladder pump can be used for normal purging and sampling (see FS 2213 and FS 2221), sampling low permeability aquifers or formations (see FS 2222) and collecting filtered groundwater samples (see FS 2225, section 1).

2.2.2.2 The bladder pump system is composed of the pump, the compressed air tubing, the water discharge tubing, the controller and a compressor or compressed gas supply.

2.2.2.3 The pump consists of a bladder and an exterior casing or pump body that surrounds the bladder and two (2) check valves. These parts can be composed of various materials, usually combinations of polyvinyl chloride (PVC), Teflon, Polyethylene, PP and stainless steel. Other materials must be compatible with the analytes of interest. See Table FS 1000-3 for restrictions.

2.2.2.4 If purging and sampling for organics:

- The pump body must be constructed of stainless steel and the valves and bladder must be Teflon, Polyethylene or PP
- The entire length of the delivery tube must be Teflon, Polyethylene or PP.
- Any cabling must be sealed in Teflon Polyethylene or PP, or be constructed of stainless steel.
- Permanently installed pumps may have a PVC pump body as long as the pump remains in contact with the water in the well.

3. BAILERS:

3.1 <u>Purging</u>: DEP does not recommend using bailers for purging unless no other equipment can be used or purging with a bailer has been specifically authorized by a DEP program, permit, contract or order (see Table FS 2200-3). Use a bailer if there is non-aqueous phase liquid (free product) in the well or non-aqueous phase liquid is suspected to be in the well. If in doubt about the appropriateness of using a bailer at a site or during a particular sampling event, contact the appropriate DEP program or project manager. If a bailer is used, follow FS 2213, section 4, with no deviations.

3.2 Sampling: Bailers may be used to routinely collect some analyte groups or under specific circumstances for other analyte groups (see Table FS 2200-3).

3.3 Construction and Type:

3.3.1 Bailers must be constructed of materials compatible with the analytes of interest. See Table FS 1000-3 for restrictions.

3.3.2 Stainless steel, Teflon, Polyethylene and PP bailers may be used to sample all analytes.

3.3.3 Use disposable bailers when sampling grossly contaminated sample sources.

3.3.4 DEP recommends using dual check valve bailers when collecting samples.

3.3.5 Use bailers with a controlled flow bottom when collecting volatile organic samples.

3.3.6 Use bailers that can be pressurized when collecting filtered samples for metals.

3.4 Contamination Prevention:

3.4.1 Keep the bailer wrapped (foil, butcher paper, etc.) until just before use.

3.4.2 Use protective gloves to handle the bailer once it is removed from its wrapping.

3.4.3 Handle the bailer by the lanyard to minimize contact with the bailer surface.

4. LANYARDS

4.1 Lanyards must be made of non-reactive, non-leachable material such as cotton twine, nylon, or stainless steel; or, coated with Teflon, Polyethylene or PP.

4.1.1 Evaluate the appropriateness of the lanyard material with analyses of equipment blanks for the analytes of interest, as necessary.

4.2 Discard cotton twine, nylon, and non-stainless steel braided lanyards after sampling each monitoring well.

4.3 Decontaminate stainless steel, coated Teflon, Polyethylene and PP lanyards between monitoring wells (see FC 1003). They do not need to be decontaminated between purging and sampling operations.

4.4 Securely fasten lanyards to downhole equipment (bailers, pumps, etc.).

4.5 Do not allow lanyards used for downhole equipment to touch the ground surface.

FS 2210. GROUNDWATER PURGING

Perform procedures in the following sections to calculate purging parameters and to purge groundwater from monitoring wells, wells with installed plumbing, high-volume wells, air stripper systems and other remedial treatment systems.

FS 2211 *Water Level and Purge Volume Determination*

Collect representative groundwater samples from the aquifer. The amount of water that must be purged from a well is determined by the volume of water and/or field parameter stabilization.

1. GENERAL EQUIPMENT CONSIDERATIONS

1.1 Selection of appropriate purging equipment depends on the analytes of interest, the well diameter, transmissivity of the aquifer, the depth to groundwater and other site conditions.

1.2 Use a pump to purge the well.

1.3 Use a bailer if there is non-aqueous phase liquid in the well or non-aqueous phase liquid is suspected to be in the well.

1.4 Bailers may be used if approved by a DEP program, or if bailer use is specified in a permit, contract or DEP order (see Table FS 2200-3). If used, bailers must be of appropriate type and construction, and the user must follow the procedure outlined in FS 2213, section 4, with no deviations. If in doubt about the appropriateness of using a bailer at a site or during a particular sampling event, contact the appropriate DEP program or project manager. DEP does not recommend using bailers because improper bailing:

1.4.1 Introduces atmospheric oxygen which precipitates metals (i.e., iron) or causes other changes in the chemistry of the water in the sample (i.e., pH)

1.4.2 Agitates groundwater which biases volatile and semi-volatile organic analyses due to volatilization

1.4.3 Agitates the water in the aquifer and resuspends fine particulate matter

1.4.4 Surges the well, loosening particulate matter in the annular space around the well screen

1.4.5 Introduces dirt into the water column if the sides of the casing wall are scraped

2. INITIAL INSPECTION

2.1 Verify the identification of the monitoring well by examining markings, sign plates, placards or other designations.

2.2 Remove the well cover and remove all standing water around the top of the well casing (manhole) before opening the well cap.

2.3 Inspect the exterior protective casing of the monitoring well for damage and document the results of the inspection if there is a problem.

2.4 It is recommended that you place a protective covering around the well head. Replace the covering if it becomes soiled or ripped.

2.5 Inspect the well lock and determine whether the cap fits tightly. Replace the cap if necessary.

3. WATER LEVEL MEASUREMENTS: Use an electronic probe or chalked tape to determine the water level.

3.1 General Procedures

Perform these steps using either the electronic probe or chalked tape method.

3.1.1 Decontaminate all equipment that will contact the groundwater in the well before use.

3.1.2 Measure the depth to groundwater from the top of well casing to the nearest 0.01 foot and always measure from the same reference point or survey mark on the well casing. If there is no reference mark, measure from the north side of the casing.

3.1.3 Record the measurement and the reference point.

3.2 Electronic Probe

3.2.1 Follow the manufacturer's instructions for use.

3.2.2 Record the measurement.

3.3 Chalked Line Method: This method is not recommended if collecting samples for organic or inorganic parameters.

3.3.1 Lower chalked tape into the well until the lower end is in the water (usually determined by the sound of the weight hitting the water).

3.3.2 Record the length of the tape relative to the reference point (see section 3.2 above).

3.3.3 Quickly remove the tape from the well.

3.3.4 Record the length of the wetted portion to the nearest 0.01 foot.

3.3.5 Determine the depth to water by subtracting the length of the wetted portion (see section 3.5.3 above) from the total length (see section 3.5.2 above). Record the result.

4. WATER COLUMN DETERMINATION

4.1 Do not determine the total depth of the well by lowering the probe to the bottom of the well immediately before purging and sampling. If the well must be sounded, delay purging and sampling activities for at least 24 hours after the well was sounded or for a time sufficient to meet the purge stabilization criterion for turbidity. Alternatively, collect samples before sounding the well.

4.2 Subtract the depth to the top of the water column from the total well depth to determine the length of the water column.

4.3 The total well depth depends on the well construction. Some wells may be drilled in areas of sinkhole or karst formations or rock leaving an open borehole. Attempt to find the total borehole depth in cases where there is an open borehole below the cased portion.

5. WELL WATER VOLUME

5.1 Calculate the total volume of water in gallons in the well using the following equation:

V = (0.041)d x d x h

Where: V = volume in gallons

d = well diameter in inches

h = height of the water column in feet

5.2 The total volume of water in the well may also be determined with the following equation by using a casing volume per foot factor (Gallons per Foot of Water) for the appropriate diameter well:

V = [Gallons per Foot of Water] x h

Where: V = volume in gallons

h = height of the water column in feet

Casing Internal Diameter	Approximate Gallons per Foot of Water
0.75”	0.02
1”	0.04
1.25”	0.06
2”	0.16
3”	0.37
4”	0.65
5”	1.02
6”	1.47
12”	5.88

5.3 Record all measurements and calculations in the field records.

6. Purging Equipment Volume

Calculate the total volume of the pump, associated tubing and container that is used for in situ measurements (flow container), if used, using the following equation:

V = p + ((0.041)d x d x l) + fc

Where: V = volume in gallons

p = volume of pump in gallons

d = tubing diameter in inches

l = length of tubing in feet

fc = volume of flow cell in gallons

7. When collecting samples from multiple wells on a site, if the groundwater elevation data are to be used to construct groundwater elevation contour maps, all water level measurements must be taken within the same 24-hour time interval unless a shorter time period is required by a DEP program. If the site is tidally influenced, complete the water level measurements within the time frame of an incoming or outgoing tide.

FS 2212 *Well Purging Techniques*

The selection of the purging technique and equipment is dependent on the hydrogeologic properties of the aquifer, especially depth to groundwater and hydraulic conductivity. The intent of proper purging is to stabilize the water level in the well and minimize the hydraulic stress to the hydrogeologic formation.

Every attempt must be made to match the pumping rate with the recharge rate of the well before evaluating the purging completion criteria.

A flowchart which summarizes purging procedure options is presented in Figure FS 2200-2.

Select equipment using the construction and configuration requirements specified in Table FS 2200-1. See the discussions in FS 2201.

1. MEASURING THE PURGE VOLUME: The volume of water that is removed during purging must be recorded. Measure the volume during the purging operation.

1.1 Collect the water in a graduated container and multiply the number of times the container was emptied by the volume of the container, or

1.2 Estimate the volume based on pumping rate. Use this technique only if the pumping rate is constant. Determine the pumping rate by measuring the amount of water that is pumped for a fixed period of time or use a flow meter.

1.2.1 Calculate the amount of water that is discharged per minute:

$$D = \frac{\text{Measured amount}}{\text{Total time in minutes}}$$

1.2.2 Calculate the time needed to purge one (1) well volume or one (1) purging equipment volume:

$$\text{Time} = \frac{V}{D}$$

Where: V = well volume determined from FS 2211, section 5, or purging equipment volume

D = discharge rate calculated in section 1.2.1. above

1.2.3 Make new measurements (see section 1.2.1 above) each time the pumping rate is changed, or

1.3 Use a totalizing flow meter.

1.3.1 Record the reading on the totalizer prior to purging.

1.3.2 Record the reading on the totalizer at the end of purging.

1.3.3 Subtract the reading on the totalizer prior to purging from the reading on the totalizer at the end of purging to obtain the volume purged.

1.4 Record in the field records the times that purging begins and ends.

2. Stabilization Measurement Frequency

2.1 Begin to record stabilization measurements after pumping the minimum volume as prescribed in options 2.3 – 2.5 below. Every attempt must be made to match the pumping rate with the recharge rate of the well before evaluating the purging criteria.

2.2 If the well screened interval is not known, use option 2.3, below.

2.3 <u>Wells with Fully Submerged Screen and Pump or Intake Tubing Placed at the Top of the Water Column (conventional purge):</u> Purge until the water level has stabilized (well recovery rate equals the purge rate), then purge a minimum of one (1) well volume prior to collecting measurements of the stabilization parameters. Allow at least one quarter (1/4) well volume to purge between subsequent measurements.

2.4 <u>Wells with Fully Submerged Screen and Pump or Intake Tubing Placed Within the Screened Interval (minimizing purge volume):</u> Purge until the water level has stabilized (well recovery rate equals the purge rate), then purge a minimum of one (1) volume of the pump, associated tubing and flow container (if used) prior to collecting measurements of the stabilization parameters. Take measurements of the stabilization parameters no sooner than two (2) minutes apart. Purge at least three (3) volumes of the pump, associated tubing and flow container, if used, prior to collecting a sample.

If the water level drops into the screened interval during purging, lower the pump or tubing intake as in FS 2213, section 1.3 below and follow purging procedures for partially submerged well screens (2.5 below).

2.5 Wells with a Partially Submerged Well Screen: Purge until the water level has stabilized (well recovery rate equals the purge rate), then purge a minimum of one (1) well volume prior to collecting measurements of the stabilization parameters. Take measurements of the stabilization parameters no sooner than two (2) minutes apart.

3. PURGING COMPLETION: DEP recommends the use of a flow-through container to measure the stabilization parameters discussed below. Alternatively, measure all parameters *in situ* by inserting measurement probes into the well at the depth appropriate for the purging option. Purging is considered complete if the criteria in section 3.1, 3.2 or 3.3 below are satisfied. Make every attempt to satisfy the criteria in section 3.1. Every attempt must be made to match the pumping rate with the recharge rate of the well before evaluating the purging criteria.

3.1 Three (3) consecutive measurements of the five (5) parameters listed below must be within the stated limits. The measurements evaluated must be the last three consecutive measurements taken before purging is stopped. The range between the highest and the lowest values for the last three measurements of temperature, pH and specific conductance cannot exceed the stated limits. The last three consecutive measurements of dissolved oxygen and turbidity must all be at or below the listed thresholds.

- Temperature: ± 0.2° C
- pH: ± 0.2 Standard Units
- Specific Conductance: ± 5.0% of reading
- Dissolved Oxygen: ≤20% Saturation
- Turbidity: ≤20 NTU

3.2 Naturally occurring conditions may prevent attaining the ≤20% saturation criterion for dissolved oxygen, typically in surficial aquifers. See section 3.5, below.

3.3 Naturally occurring conditions may prevent attaining the ≤20 NTU criterion for turbidity. However, when collecting groundwater samples for metals or certain inorganic (e.g., phosphorus forms) or extractable organic (e.g. polynuclear aromatic hydrocarbons) chemicals, make every attempt to reduce turbidity to ≤20 NTU to avoid a potential turbidity-associated bias for these analytes. See section 3.5, below.

3.4 Document and report the following, as applicable, except that the last four (4) items only need to be submitted once:

- Purging rate.
- Drawdown in the well, if any.
- Pump or tubing intake placement.
- Length and location of the screened interval.
- A description of the process and the data used to design the well.
- The equipment and procedure used to install the well.

- The well development procedure.
- Pertinent lithologic or hydrogeologic information.

3.5 If the criteria in section 3.1 above for dissolved oxygen and/or turbidity cannot be met, then three (3) consecutive measurements of the five (5) parameters listed below must be within the stated limits.

3.5.1 The measurements evaluated must be the last three consecutive measurements taken before purging is stopped. The range between the highest and the lowest values for the last three measurements cannot exceed the stated limits.

- Temperature: ± 0.2° C
- pH: ± 0.2 Standard Units
- Specific Conductance: ± 5.0% of reading
- Dissolved Oxygen: ± 0.2 mg/L or 10%, whichever is greater
- Turbidity: ± 5 NTUs or 10%, whichever is greater

3.5.2 Additionally, document and report the following, as applicable, except that the last four (4) items only need to be submitted once:

- Purging rate.
- Drawdown in the well, if any.
- Pump or tubing intake placement.
- Length and location of the screened interval.
- A description of conditions at the site that cause the dissolved oxygen to be high and/or dissolved oxygen measurements made within the screened or open borehole portion of the well with a downhole dissolved oxygen probe.
- A description of conditions at the site that cause the turbidity to be high and any procedures that will be used to minimize turbidity in the future.
- A description of the process and the data used to design the well.
- The equipment and procedure used to install the well.
- The well development procedure.
- Pertinent lithologic or hydrogeologic information.

3.5.3 If from review of the submitted data the Department determines that both the elevated Dissolved Oxygen and Turbidity measurements are due to naturally occurring conditions, then only the first four (4) items are required to be submitted in future reports. However, if the Department cannot determine if the Dissolved Oxygen or Turbidity is elevated due to naturally occurring conditions, then in addition to the first four (4) items, a description of the conditions at the site that caused the affected parameter(s) to be high is required to be submitted in future reports.

3.6 If the stabilization parameters in either section 3.1 or 3.2 cannot be met, and all attempts have been made to minimize the drawdown, check the instrument condition and calibration, purging flow rate and all tubing connections to determine if they might be affecting the ability to achieve stable measurements. All measurements that were made during the attempt must be documented. The sampling team leader may decide whether or not to collect a sample or to continue purging after five (5) well volumes (conventional purge section 2.1 or 2.3 above) or five (5) volumes of the screened interval (minimizing purge volumes in section 2.2 above).

Further, the report in which the data are submitted must include the following, as applicable, except that the last four (4) items only need to be submitted once:

- Purging rate.
- Pump or tubing intake placement.
- Length and location of the screened interval.
- Drawdown in the well, if any.
- A description of conditions at the site that caused the dissolved oxygen to be high and/or dissolved oxygen measurements made within the screened or open borehole portion of the well with a downhole dissolved oxygen probe.
- A description of conditions at the site that caused the turbidity to be high and any procedures that will be used to minimize turbidity in the future.
- A description of the process and the data used to design the well.
- The equipment and procedure used to install the well.
- The well development procedure.
- Pertinent lithologic or hydrogeologic information.

If from review of the submitted data the DEP determines that both the elevated Dissolved Oxygen and Turbidity measurements are due to naturally occurring conditions, then only the first four (4) items are required to be submitted in future reports. However, if the DEP cannot determine if the Dissolved Oxygen or Turbidity is elevated due to naturally occurring conditions, then in addition to the first four (4) items, a description of the conditions at the site that caused the affected parameter(s) to be high is required to be submitted in future reports.

3.7 One fully dry purge (not recommended). This criterion applies only if purging was attempted per FS 2212, FS 2213, and section 3.4.1 below, and if it is impossible to balance the pumping rate with the rate of recharge at very low pumping rates (< 100 mL/minute).

3.7.1 If wells have previously and consistently purged dry, when purged according to FS 2212 and FS 2213, and the current depth to groundwater indicates that the well will purge dry during the current sampling event, minimize the amount of water removed from the well by using the same pump to purge and collect the sample:

3.7.1.1 Place the pump or tubing intake within the well screened interval.

3.7.1.2 Use very small diameter Teflon, Polyethylene or PP tubing and the smallest possible pump chamber volume to minimize the total volume of water pumped from the well and to reduce drawdown.

3.7.1.3 Select tubing that is thick enough to minimize oxygen transfer through the tubing walls while pumping.

3.7.1.4 Pump at the lowest possible rate (100 mL/minute or less) to reduce drawdown to a minimum.

3.7.1.5 Purge at least two (2) volumes of the pumping system (pump, tubing and flow cell, if used).

3.7.1.6 Measure pH, Specific Conductance, Temperature, Dissolved Oxygen and Turbidity and begin to collect the samples (see FS 2222).

4. Collect samples immediately after purging is complete.

4.1 The time period between completing the purge and sampling cannot exceed six (6) hours.

4.2 If sample collection does not occur within one (1) hour of purging completion, re-measure the five (5) field parameters Temperature, pH, Specific Conductance, Dissolved Oxygen and Turbidity just prior to collecting the sample.

4.2.1 If the measured values are not within 10 percent of the previous measurements, re-purge the well.

4.2.2 See section 3.4 above when collecting samples from wells that have purged dry.

FS 2213 *Purging Wells Without Plumbing (Monitoring Wells)*

1. TUBING/PUMP PLACEMENT

1.1 Do not lower the pump or intake hose (tubing) to the bottom of the well. Pump or tubing placement procedures will be determined by the purging option selected in FS 2212, section 2 above or FS 2214 below.

1.1.1 Minimizing Purge Volume: If the following conditions can be met, position the intake hose (tubing) or pump in the screened or open borehole interval.

- The same pump must be used for both purging and sampling,
- The well screen or borehole interval must be less than or equal to 10 feet, and
- The well screen or borehole must be fully submerged.

1.1.2 If the position or length of the screened interval or open borehole is unknown or estimated, place the intake hose (tubing) or pump to perform conventional purging in 1.2 below.

1.1.3 Position the pump or intake hose when purging large-diameter deep wells with open boreholes using the procedure in FS 2214 below.

1.2 Conventional Purging: Position the pump or intake tubing in the top one foot of the water column or no deeper than necessary for the type of pump.

1.2.1 If purging with a bailer, see section 4 below.

1.3 Partially Submerged Screened Interval: If the well screen or open borehole is partially submerged, and the pump will be used for both purging and sampling, position the pump or intake hose (tubing) in the portion of the water column within the submerged screened or open borehole interval.

1.3.1 If the position or length of the screened interval or open borehole is unknown or estimated, place the intake hose (tubing) or pump to perform conventional purging in 1.2 above.

1.3.2 Purge large-volume, high-recharge wells as in FS 2214 below.

1.3.3 If purging with a bailer, see section 4 below.

2. NON-DEDICATED (PORTABLE) PUMPS

2.1 Variable Speed Peristaltic Pump

2.1.1 Install a new, 1-foot maximum length of silicone tubing in the peristaltic pump head.

2.1.2 Attach a short section of tubing to the discharge side of the pump-head silicone tubing and into a graduated container.

2.1.3 Attach one end of a length of new or precleaned transport tubing to the intake side of the pump head silicone tubing.

2.1.4 Place the transport tubing in the monitoring well per one of the options in FS 2213, section 1 above.

2.1.5 Measure the depth to groundwater at frequent intervals.

2.1.6 Record these measurements.

2.1.7 Adjust the purging rate so that it is equivalent to the well recovery rate to minimize drawdown.

2.1.8 If the purging rate exceeds the well recovery rate, reduce the pumping rate to balance the withdrawal rate with the recharge rate.

2.1.9 If the water table continues to drop during pumping, lower the tubing at the approximate rate of drawdown so that the water is removed from the top of the water column.

2.1.10 Record the purging rate each time the rate changes.

2.1.11 Measure the purge volume by one of the methods outlined in FS 2212, section 1.

2.1.12 Record this measurement.

2.1.13 Decontaminate the pump and tubing between wells (see FC 1000) or only the pump if precleaned tubing is used for each well.

2.2 Variable Speed Centrifugal Pump

2.2.1 Position fuel powered equipment **downwind** and at least 10 feet from the well head. Make sure that the exhaust faces downwind.

2.2.2 Place the decontaminated suction hose so that water is always pumped from the top of the water column.

2.2.3 Equip the suction hose with a foot valve to prevent purge water from re-entering the well.

2.2.4 Measure the depth to groundwater at frequent intervals.

2.2.5 Record these measurements.

2.2.6 Adjust the purging rate so that it is equivalent to the well recovery rate to minimize drawdown.

2.2.7 If the purging rate exceeds the well recovery rate, reduce the pumping rate to balance the withdrawal rate with the recharge rate.

2.2.8 If the water table continues to drop during pumping, lower the tubing at the approximate rate of drawdown so that the water is removed from the top of the water column.

2.2.9 Record the purging rate each time the rate changes.

2.2.10 Measure the purge volume by one of the methods outlined in FS 2212, section 1.

2.2.11 Record this measurement.

2.2.12 Decontaminate the pump and tubing between wells (see FC 1000) or only the pump if precleaned tubing is used for each well.

2.3 <u>Variable Speed Electric Submersible Pump</u>

2.3.1 Position fuel powered equipment downwind and at least 10 feet from the well head. Make sure that the exhaust faces downwind.

2.3.2 Carefully position the decontaminated pump per one of the options in FS 2213, section 1 above.

2.3.3 Measure the depth to groundwater at frequent intervals.

2.3.4 Record these measurements.

2.3.5 Adjust the purging rate so that it is equivalent to the well recovery rate to minimize drawdown.

2.3.6 If the purging rate exceeds the well recovery rate, reduce the pumping rate to balance the withdrawal rate with the recharge rate.

2.3.7 If the water table continues to drop during pumping, lower the tubing or pump at the approximate rate of drawdown so that the water is removed from the top of the water column.

2.3.8 Record the purging rate each time the rate changes.

2.3.9 Measure the purge volume by one of the methods outlined in FS 2212, section 1.

2.3.10 Record this measurement.

2.3.11 Decontaminate the pump and tubing between wells (see FC 1000) or only the pump if precleaned tubing is used for each well.

2.4 Variable Speed Bladder Pump

2.4.1 Position fuel powered equipment **downwind** and at least 10 feet from the well head. Make sure that the exhaust faces downwind.

2.4.2 Attach the tubing and carefully position the pump per one of the options in FS 2213, section 1 above.

2.4.3 Measure the depth to groundwater at frequent intervals.

2.4.4 Record these measurements.

2.4.5 Adjust the purging rate so that it is equivalent to the well recovery rate to minimize drawdown.

2.4.6 If the purging rate exceeds the well recovery rate, reduce the pumping rate to balance the withdrawal rate with the recharge rate.

2.4.7 If the water table continues to drop during pumping, lower the tubing or pump at the approximate rate of drawdown so that the water is removed from the top of the water column.

2.4.8 Record the purging rate each time the rate changes.

2.4.9 Measure the purge volume by one of the methods outlined in FS 2212, section 1.

2.4.10 Record this measurement.

2.4.11 Decontaminate the pump and tubing between wells (see FC 1000) or only the pump if precleaned tubing is used for each well.

3. DEDICATED PORTABLE PUMPS: Place dedicated pumps per one of the options in FS 2213, section 1 above.

3.1 Variable Speed Electric Submersible Pump

3.1.1 Position fuel powered equipment **downwind** and at least 10 feet from the well head. Make sure that the exhaust faces downwind.

3.1.2 Measure the depth to groundwater at frequent intervals.

3.1.3 Record these measurements.

3.1.4 Adjust the purging rate so that it is equivalent to the well recovery rate to minimize drawdown.

3.1.5 If the purging rate exceeds the well recovery rate, reduce the pumping rate to balance the withdrawal with the recharge rate.

3.1.6 Record the purging rate each time the rate changes.

3.1.7 Measure the purge volume by one of the methods outlined in FS 2212, section 1.

3.1.8 Record this measurement.

3.2 Variable Speed Bladder Pump

3.2.1 Position fuel powered equipment **downwind** and at least 10 feet from the well head. Make sure that the exhaust faces downwind.

3.2.2 Measure the depth to groundwater at frequent intervals.

3.2.3 Record these measurements.

3.2.4 Adjust the purging rate so that it is equivalent to the well recovery rate to minimize drawdown.

3.2.5 If the purging rate exceeds the well recovery rate, reduce the pumping rate to balance the withdrawal with the recharge rate.

3.2.6 Record the purging rate each time the rate changes.

3.2.7 Measure the purge volume by one of the methods outlined in FS 2212, section 1.

3.2.8 Record this measurement.

4. BAILERS: DEP recommends against using bailers for purging except as a last contingency, or if free product is present in the well or suspected to be in the well. However, they may be used if approved by a DEP program, or specified in a permit, contract or DEP order (see Table FS 2200-3 and FS 2211, section 1.3). If in doubt about the appropriateness of using a bailer at a site or during a particular sampling event, contact the appropriate DEP program or project manager.

4.1 Minimize handling the bailer as much as possible.

4.1.1 Remove the bailer from its protective wrapping just before use.

4.1.2 Attach a lanyard of appropriate material (see FS 2201, section 4).

4.1.3 Use the lanyard to move and position the bailer.

4.2 Lower and retrieve the bailer slowly and smoothly.

4.3 Lower the bailer carefully into the well to a depth approximately a foot above the water column.

4.3.1 Do not lower the top of the bailer more than one (1) foot below the top of the water table so that water is removed from the top of the water column. Ensure that the length of the bailer does not exceed the length of the water column.

4.3.2 Allow time for the bailer to fill with aquifer water as it descends into the water column.

4.4 Carefully raise the bailer.

4.4.1 Retrieve the bailer at the same rate of 2 cm/sec until the bottom of the bailer has cleared to top of the water column.

4.5 Measure the purge volume by one of the methods outlined in FS 2212, section 1.

4.5.1 Record the volume of the bailer.

4.6 Continue to carefully lower and retrieve the bailer as described above until the purging completion conditions specified in FS 2212, section 3, have been satisfied.

4.6.1 Remove at least one (1) well volume before collecting measurements of the field parameters. Take each subsequent set of measurements after removing at least one quarter (1/4) well volume between measurements.

FS 2214 *Purging Large-Volume, High-Recharge Wells With Portable Pumps*

If a well originally constructed for high-flow-rate pumping will be sampled as a monitoring well, use these guidelines to develop a purging procedure applicable to the specific details of the well construction. Typical wells constructed for this purpose may be deep, large-diameter wells with a section of open borehole. Evaluate each well on a case-by-case basis and consider any available information on the construction and hydraulic performance of the well.

1. PURGING PROCEDURE

1.1 Place the pump at the top of the open borehole segment of the well.

1.2 Start purging while monitoring stabilization parameters as in FS 2212, section 3 above.

1.3 Purge at least one equipment volume before measuring stabilization parameters.

1.4 If the well is being purged for the first time using these guidelines, monitor stabilization parameters for an extended period until confident that sufficient volume has been pumped from the open borehole to draw fresh formation water into the pump tubing and flow-through container. Use the information obtained from the first-time purging of the well to determine the pumping rate and duration of purging required for future sampling events at the well.

1.5 Purge at least three equipment volumes before evaluating purging completion.

2. PURGING COMPLETION

2.1 Complete the purging of the well when the last three consecutive measurements of the purge stabilization parameters have met the applicable criteria specified in FS 2212, section 3 above.

3. Collect samples from the well using the procedures in FS 2221, section 1 below.

FS 2215. *Purging Wells With Plumbing (production wells or permanently installed pumps equipped with sampling ports or sampling spigots)*

Wells with in-place plumbing are commonly found at municipal water treatment plants, industrial water supplies, private residences, etc. Depending on the sampling objective for collecting samples using installed plumbing, purge the system and collect samples closest to the point of consumption, or, as close to the source well as possible. When purging is required and the purge volume of the plumbing system is not known, purge the system until the purging completion criteria in FS 2212, section 3, have been met.

1. CONTINUOUSLY RUNNING PUMPS

1.1 Select the spigot that is closest to the pump and before any storage tanks (if possible).

1.2 Remove all hoses, aerators and filters (if possible).

1.3 Open the spigot and purge at maximum flow.

1.4 If a storage tank is located between the pump and the spigot, purge the volume of the tank, lines and spigot.

1.5 If the spigot is before any storage tank, purge until sufficient volume is removed to flush the stagnant water from the spigot and the tap line to the spigot.

1.6 Reduce the flow rate to ≤ 500 mL/minute (a 1/8" stream) or approximately 0.1 gal/minute before collecting samples. When sampling for volatile organic compounds, reduce the flow to ≤100 mL/minute before collecting the samples.

2. INTERMITTENTLY RUNNING PUMPS

2.1 Select the spigot that is closest to the pump and before any storage tanks (if possible).

2.2 Remove all hoses, aerators and filters (if possible).

2.3 Open the spigot and purge sufficient volume at a maximum, practical flow rate to flush the spigot and lines and until the purging completion criteria in FS 2212, section 3, have been met.

2.4 If a storage tank is located between the pump and the spigot, purge the volume of the tank, lines and spigot.

2.5 Ensure that the purge stabilization measurement of dissolved oxygen is not biased with aeration of the sample by a high flow rate in the flow-through container.

2.6 Reduce the flow rate to ≤ 500 mL/minute (a 1/8" stream) or approximately 0.1 gal/minute before collecting samples. When sampling for volatile organic compounds, reduce the flow to ≤ 100 mL/minute before collecting the samples.

FS 2216. *Purging Airstrippers and Remedial Treatment Systems*

If collecting samples for groundwater contamination monitoring, follow FS 2215above.

FS 2220. GROUNDWATER SAMPLING TECHNIQUES

1. Purge wells using the techniques outlined in FS 2210.
2. Replace the protective covering around the well if it is soiled or torn after completing the purging operations.
3. EQUIPMENT CONSIDERATIONS

Follow all notes and restrictions as indicated in Table FS 2200-1 and as discussed in FS 2201.

NOTE: The only pumps that are currently approved for use in collecting volatile organic samples through the pump are stainless steel and Teflon variable speed submersible pumps, stainless steel and Teflon or Polyethylene variable speed bladder pumps, and permanently installed PVC bodied pumps (variable speed bladder or submersible pumps) as long as the pump remains in contact with the water in the well at all times.

3.1 Collect the sample into the sample container from the sampling device. **Do not** use intermediate containers.

3.2 In order to avoid contaminating the sample or loss of analytes from the sample:

3.3 Handle the sampling equipment as little as possible.

3.3.1 Minimize the equipment that is exposed to the sample.

3.3.2 Minimize aeration of samples collected for VOC analysis.

3.3.3 Reduce sampling pump flow rates to $\leq$ 100 mL/minute when collecting VOC samples.

3.4 Dedicated Sampling Equipment

3.4.1 Whenever possible, use dedicated equipment because it significantly reduces the chance of cross-contamination.

3.4.2 Dedicated is defined as equipment that is to be used solely for one location for the life of that equipment (e.g., permanently mounted pump).

3.4.3 All material construction and restrictions from Table FS 2200-1 also apply to dedicated equipment. Purchase equipment with the most sensitive analyte of interest in mind.

3.5 Cleaning/Decontamination

3.5.1 Clean or ensure dedicated pumps are clean before installation. They do not need to be cleaned prior to each use but must be cleaned if they are withdrawn for repair or servicing.

3.5.2 Clean or make sure any permanently mounted tubing is clean before installation.

3.5.3 Change or clean tubing when the pump is withdrawn for servicing.

3.5.4 Clean any replaceable or temporary parts as specified in FC 1000.

3.5.5 Collect equipment blanks on dedicated pumping systems when the tubing is cleaned or replaced.

3.5.6 Clean or ensure dedicated bailers are clean before placing them into the well.

3.5.7 Collect an equipment blank on dedicated bailers before introducing them into the water column.

3.5.8 Suspend dedicated bailers above the water column if they are stored in the well.

FS 2221. *Sampling Wells Without Plumbing*

1. SAMPLING WITH PUMPS: Variable speed stainless steel and Teflon submersible pumps and stainless steel, Teflon or Polyethylene bladder pumps, and permanently installed PVC-bodied variable speed submersible or bladder pumps, as long as the pump remains in contact with the water in the well at all times, may be used to sample for all organics. The delivery tubing must be Teflon, Polyethylene or PP. **Extractable organics** may be collected through a peristaltic pump if ≤ 1 foot of silicone tubing is used in the pump head or a vacuum trap is used (see Figure FS 2200-1 for specific configuration). Follow all notes and restrictions as defined in Table FS 2200-1 and discussed in Equipment and Supplies (FS 2201) when using pumps to collect samples.

Do not lower the pump or tubing to the bottom of the well.

1.1 <u>Peristaltic Pump</u>

1.1.1 <u>Volatile Organics Using Manual Fill and Drain Method</u>: Collect volatile organics last. If the pump tubing is placed within the screened interval, the tubing cannot be reinserted into the well, and steps 1.1.1.3 through 1.1.1.6 below are prohibited.

1.1.1.1 Ensure that there is sufficient tubing volume to fill the requisite number of VOC vials.

1.1.1.2 Remove the drop tubing from the inlet side of the pump.

1.1.1.3 Submerse the drop tubing into the water column and allow it fill.

1.1.1.4 Remove the drop tubing from the well.

1.1.1.5 Prevent the water in the tubing from flowing back into the well.

1.1.1.6 Carefully allow the groundwater to drain by gravity into the sample vials. Avoid turbulence. Do not aerate the sample. The flow rate must be $\leq$ 100 mL/minute.

1.1.1.7 Repeat steps 1.1.1.3 - 1.1.1.6 until enough vials are filled.

1.1.2 <u>Volatile Organics Using the Pump to Fill and Drain the Tubing:</u> Collect volatile organics last. If the pump tubing is placed within the screened interval, the tubing cannot be reinserted into the well, and steps 1.1.2.2 through 1.1.2.8 below are prohibited.

1.1.2.1 Ensure that there is sufficient tubing volume to fill the requisite number of VOC vials.

1.1.2.2 Submerse the drop tubing into the water column.

1.1.2.3 Use the pump to fill the drop tubing.

1.1.2.4 Quickly remove the tubing from the pump.

1.1.2.5 Prevent the water in the tubing from flowing back into the well.

1.1.2.6 Remove the drop tubing from the well and fill the vials using the pump or gravity-drain methods in steps 1.1.2.7 or 1.1.2.8 below.

1.1.2.7 Reverse the flow on the peristaltic pump to deliver the sample into the vials at a slow, steady rate. The flow rate must be $\leq$ 100 mL/minute.

1.1.2.8 Or, remove the drop tubing from the inlet side of the pump and carefully allow the groundwater to drain into the sample vials. Avoid turbulence. Do not aerate the sample. The flow rate must be $\leq$ 100 mL/minute.

1.1.2.9 Repeat steps 1.1.2.2 through 1.1.2.8 until enough vials are filled.

1.1.3 Extractable Organics Collected Through Silicone Pump-Head Tubing:

1.1.3.1 Ensure that a 1-foot maximum length of new silicone tubing was installed in the peristaltic pump head assembly before the well was purged if the same pump is being used to purge and sample the well. Otherwise, install a new length of tubing as described above.

1.1.3.2 Collect extractable organic samples directly from the effluent delivery tubing (attached to discharge side of the silicone pump head tubing) into the sample container.

1.1.3.3 If there is a concern that sample analytes are absorbed, adsorbed, leached or otherwise affected or lost by pumping through the silicone pump-head tubing, sample the well using the organic trap assembly in 1.1.4 below.

1.1.4 Extractable Organics Using an Optional Organic Trap Assembly

1.1.4.1 Assemble the components of the pump and trap according to Figure FS 2200-1.

1.1.4.2 The sample container should be the trap bottle.

1.1.4.3 All equipment that contacts the groundwater **before** the sample container must be constructed of Teflon, Polyethylene, PP, stainless steel or glass, including the transport tubing to and from the sample container, the interior liner of the container cap and all fittings. **Do not use a rubber stopper as a cap.**

1.1.4.4 Connect the outflow tubing from the container to the influent side of the peristaltic pump.

1.1.4.5 Prevent the water in the down-hole delivery tubing from flowing back into the well while performing this connection.

1.1.4.6 Turn the pump on and reduce the flow rate to a smooth and even flow.

1.1.4.7 Discard a small portion of the sample to allow an air space.

1.1.4.8 Preserve (if required), label and complete the field notes.

1.1.5 Inorganics

1.1.5.1 Inorganic samples may be collected from the effluent tubing.

1.1.5.2 If samples are collected from the pump, decontaminate all tubing (including the tubing in the head) or change it between wells.

1.1.5.3 Preserve (if required), label and complete field notes.

1.2 Variable Speed Bladder Pump

1.2.1 If sampling for organics the pump body must be constructed of stainless steel and the valves and bladder must be Teflon. All tubing must be Teflon, Polyethylene, or PP and any cabling must be sealed in Teflon, Polyethylene or PP, or made of stainless steel.

1.2.2 After purging to a smooth even flow, reduce the flow rate.

1.2.3 When sampling for volatile organic compounds, reduce the flow rate to 100 mL/minute or less, if possible.

1.3 Variable Speed Submersible Pump

1.3.1 The housing must be stainless steel.

1.3.2 If sampling for organics, the internal impellers, seals and gaskets must be constructed of stainless steel, Teflon, Polyethylene or PP. The delivery tubing must be Teflon, Polyethylene or PP and the electrical cord must be sealed in Teflon and any cabling must be sealed in Teflon or constructed of stainless steel.

1.3.3 After purging to a smooth even flow, reduce the flow rate.

1.3.4 When sampling for volatile organic compounds, reduce the flow rate to 100 mL/minute or less, if possible.

2. SAMPLING WITH BAILERS: A high degree of skill and coordination are necessary to collect representative samples with a bailer. When properly used, bailers may be used to collect samples for certain analyte groups and under specific conditions (see Table FS 2200-3). They must be of an appropriate type and construction (see FS 2201, section 3), and must be used as outlined below. If in doubt about the appropriateness of using a bailer at a site or during a particular sampling event, contact the appropriate DEP program or project manager.

2.1 General Considerations

2.1.1 Minimize handling the bailer as much as possible.

2.1.1.1 Wear sampling gloves.

2.1.1.2 Remove the bailer from its protective wrapping just before use.

2.1.1.3 Attach a lanyard of appropriate material (see FS 2201, section 4).

2.1.1.4 Use the lanyard to move and position the bailers.

2.1.2 Do not allow the bailer or lanyard to touch the ground.

2.1.3 Rinsing

2.1.3.1 If the bailer is certified precleaned, no rinsing is necessary.

2.1.3.2 If both a pump and a bailer are to be used to collect samples, rinse the exterior and interior of the bailer with sample water from the pump before removing the pump.

2.1.3.3 If the purge pump is not appropriate for collecting samples (e.g., non-inert components), rinse the bailer with by collecting a single bailer of the groundwater to be sampled. Use the technique described in section 2.2, Bailing Technique, below.

2.1.3.4 Discard the water appropriately.

2.1.3.5 **Do not** rinse the bailer if Oil & Grease, TRPHs, etc., (see FS 2006) are to be collected.

2.2 Bailing Technique

2.2.1 Collect all samples that are required to be collected with a pump before collecting samples with the bailer.

2.2.2 Raise and lower the bailer gently to minimize stirring up particulate matter in the well and the water column which can increase sample turbidity.

2.2.3 Lower the bailer carefully into the well to a depth approximately a foot above the water column. Ensure that the length of the bailer does not exceed the length of the water column.

2.2.3.1 When the bailer is in position, lower the bailer into the water column at a rate of 2 cm/sec until the desired depth is reached (see section 2.2.3 above).

2.2.4 Do not lower the top of the bailer more than one (1) foot below the top of the water table so that water is removed from the top of the water column.

2.2.5 Allow time for the bailer to fill with aquifer water as it descends into the water column.

2.2.6 Do not allow the bailer to touch the bottom of the well or particulate matter will be incorporated into the sample.

2.2.6.1 Carefully raise the bailer (see section 2.2.2 above). Retrieve the bailer at the same rate of 2 cm/sec until the bottom of the bailer has cleared to top of the water column.

2.2.7 Lower the bailer to approximately the same depth each time.

2.2.8 Collect the sample.

2.2.8.1 Install a device to control the flow from the bottom of the bailer and discard the first few inches of water. Reduce the flow to $\leq$ 100 mL/minute when collecting VOC samples.

2.2.8.2 Fill the appropriate sample containers by allowing the sample to slowly flow down the side of the container. Minimize aeration of VOC samples.

2.2.8.3 Discard the last few inches of water in the bailer.

2.2.9 Repeat steps 2.2.1 through 2.2.8.3 for additional samples.

2.2.10 Measure the DO, pH, temperature, turbidity and specific conductance after the final sample has been collected.

2.2.10.1 Record all measurements and note the time that sampling was completed.

3. SAMPLING WELLS WITH FLOATING NON-AQUEOUS PHASE LIQUID: DEP does not recommend the sampling of wells with floating non-aqueous phase liquid for trace contaminants. This concerns primarily petroleum related sites, but includes any chemical product (e.g., solvent) that floats on the water table. Sampling is acceptable if the information is to be used for the purpose of remedial design.

Sample data from such wells cannot provide useful information regarding the level of contamination. Furthermore, these wells typically do not provide legitimate data because of permanent chemical contamination from product contact with the well casing for an extended period of time.

DEP does reserve the right to require sampling of these wells, not for levels of trace contaminants, but for confirmation of an appropriate remediation technique. This type of sampling is performed **below** the non-aqueous phase layer (see section 3.2 below).

3.1 Non-Aqueous Phase Liquid Sampling: Non-aqueous phase liquid may be evident in a cased monitoring well or in an open excavation.

3.1.1 Non-aqueous phase liquid is normally sampled for two reasons:

- Documentation for its existence and thickness; and
- Determination of the type of product so that the proper analyses can be performed to determine extent. This is only feasible for relatively recent releases as it may not be possible to identify weathered product.

3.1.2 Disposable plastic (acrylic, clear PVC) bailers are recommended for sampling. Disposable Polyethylene and PP bailers are also acceptable. Other wide mouth vessels may be used for sampling non-aqueous phase liquid in an excavation.

3.1.3 Monitoring Well

3.1.3.1 If a non-aqueous phase liquid is identified in a monitoring well during the water level measurement, measure its thickness in the well. If the thickness of the non-aqueous phase liquid is greater than 0.01 foot or product globules are present, collect a sample using a precleaned disposable bailer.

3.1.3.2 Measure the product thickness to the nearest 0.01 foot after withdrawing the bailer.

3.1.3.3 Pour a portion of the product into a glass sample container.

3.1.3.4 This sample is considered a concentrated waste. Therefore, package the container in protective wrapping to prevent breakage, isolate from other samples, and ice to 4°C.

3.1.4 Excavation

3.1.4.1 If non-aqueous phase liquid is observed in an open excavation, a glass sample container or a precleaned intermediate vessel may be used to collect the sample.

3.1.4.2 Securely tie a lanyard to the container and lower it into the excavation.

3.1.4.3 Gently lower and retrieve the container so that no solid material is released or collected.

3.1.4.4 If sufficient water is available, a bailer can be used.

3.1.4.5 Although not recommended, screened casing can be placed (or augered and placed) in the bottom of the excavation and the product sampled with a bailer.

3.1.4.6 Avoid dangerous situations, such as standing too close to the edge of an excavation, riding in the backhoe bucket, or entering a trench or excavation that may collapse.

3.1.4.7 Follow all applicable OSHA regulations.

3.2 Sampling Below Product

3.2.1 This type of depth-specific sampling to attempt to sample the dissolved constituents in the water column below the product layer is performed only at the request of DEP or its designee.

3.2.2 These data provide information that helps define adequate groundwater treatment. Without these data, incorrect (and sometimes unnecessarily expensive) remediation techniques may be designed for a situation where they are not required.

3.2.3 There are some substantial logistical problems involved with sending a sampler through non-aqueous phase liquid to sample the groundwater below. Although there are some products designed specifically for this type of sampling, they are expensive and the results may not be commensurate with their cost. The use of "self-engineered" equipment or coverings may be the best option.

3.2.4 These data are only to be used for qualitative use and will aid in deciding on an appropriate remediation technique.

3.2.5 Wrapping bailers and tubing in plastic seems to be the most popular technique in getting past the product layer.

3.2.6 Although not recommended, some have wrapped submersible pumps in several layers of plastic and retrieved each layer by a separate lanyard. One suggestion would be to use a rigid piece of stainless steel tubing wrapped in plastic.

3.2.6.1 Once the covered tubing is past the layer, pull up on the plastic, piercing the plastic and exposing the (somewhat) clean tubing inlet.

3.2.6.2 Introduce the wrapped hose slowly to not entrain any more product into the dissolved layer located below.

3.2.6.3 Also, perform this procedure with a peristaltic pump or a vacuum pump linked to a trap bottle. To use this setup, the water table must be no deeper than 15-20 feet, realizing that actual sampling may be occurring several feet below the product layer.

FS 2222. *Sampling Low Permeability Aquifers or Wells That Have Purged Dry*

1. Collect the sample(s) after the well has been purged according to FS 2212, section 3.4. Minimize the amount of water removed from the well by using the same pump to purge and collect the sample. If the well has purged dry, collect samples as soon as sufficient sample water is available.

2. Measure the five (5) field parameters Temperature, pH, Specific Conductance, Dissolved Oxygen and Turbidity at the time of sample collection.

3. Advise the analytical laboratory and the client that the usual amount of sample for analysis may not be available.

FS 2223. *Sampling Wells With In-Place Plumbing*

1. If a storage tank is present, locate a cold water spigot, valve or other sampling point close to the well head between the pump and the storage tank. If there is no sampling location between the pump and the storage tank, locate the spigot, valve or other sampling point closest to the tank.

1.1 Depending on the sampling objective for collecting samples using installed plumbing, purge the system and collect samples closest to the point of consumption, or, as close to the source well as possible.

2. Remove all screens or aerators and reduce the flow rate to no more than 500 mL/minute. If collecting samples for volatile organic compounds, reduce the flow rate to 100 mL/minute or less. Collect the samples directly into the appropriate containers.

FS 2224. *Sampling Airstripper and Remedial Treatment System Sampling*

1. Reduce the flow rate to less than 500 mL/minute and begin sample collection.
2. If collecting samples for volatile organic compounds, reduce the flow rate to 100 mL/minute or less.
3. Collect the samples directly into the appropriate containers.

FS 2225. *Filtering Groundwater Samples*

Filtered groundwater samples can only be collected after approval from the DEP program or project manager. If filtering is approved, the DEP program or permit condition may require both filtered and unfiltered samples to be collected, analyzed and reported.

1. FILTERING GROUNDWATER FOR METALS:

1.1 Unless specified otherwise by the DEP program, use a new, disposable, high capacity, 1-μm in-line filter.

1.2 Use a variable speed peristaltic, bladder or submersible pump with the in-line filter fitted on the outlet end.

1.2.1 Peristaltic pumps, bladder pumps or submersible pumps can be used when water levels are no greater than 20 to 25 feet deep.

1.2.2 Bladder pumps or submersible pumps must be used when water levels are greater than 20 to 25 feet deep.

1.3 Ensure that a 1-foot maximum length of new, silicone tubing was installed in the peristaltic pump head assembly before the well was purged if the same pump is being used to purge and sample the well. Otherwise, install a new length of tubing as described above.

1.4 Ensure that new or precleaned delivery tubing was assembled with the peristaltic pump before the well was purged if the same pump is being used to purge and sample the well. Otherwise, assemble the pump with new or precleaned delivery tubing and the new filter.

1.5 Insert the filter on the high pressure side (i.e., on the delivery side) of the pump.

1.5.1 Flush the filter before attaching to the pump tubing assembly with 30-50 mL of analyte free water or an inert gas (nitrogen) to remove atmospheric oxygen;

1.5.2 Or, with the filter attached to the pump tubing assembly, hold the filter upright with the inlet and outlet in the vertical position and pump water from the aquifer through the filter until all atmospheric oxygen has been removed.

1.6 Collect the filtered samples directly into the sample container from the high-pressure (delivery) side of the pump tubing assembly.

1.6.1 Collect filtered samples by either of the methods in 1.6.1.3 or 1.6.1.4 below if the static water level in the well is too deep for a variable speed peristaltic pump and a variable speed electric submersible pump or variable speed bladder pump is not available.

1.6.1.1 Do not agitate the sample or expose it to atmospheric oxygen.

1.6.1.2 **Do not** pour the sample into any intermediate vessel for subsequent filtration.

1.6.1.3 Collect the sample in a Polyethylene, Teflon or PP bailer that can be pressurized. When the bailer has been retrieved, immediately connect the filter and begin to pressurize the bailer;

1.6.1.4 Or, collect the sample with a bailer and immediately place the intake tube of the peristaltic pump into the full bailer and begin pumping the water through the filter as described in section 1.2 above.

1.7 **Do not** use the following equipment for filtering groundwater samples for metals:

1.7.1 Any pump and apparatus combination in which the filter is on the vacuum (suction) side of the pump.

1.7.2 Any type of syringe or barrel filtration apparatus.

1.7.3 Any filter that is not encased in a one-piece, molded unit.

2. Filtering groundwater for non-metallic analytes

2.1 The following analytes cannot be filtered:

- Oil and Grease
- Total Recoverable Petroleum Hydrocarbons (TRPH)
- FL-PRO
- Volatile Organic Compounds (VOC)
- Microbiological Analytes
- Volatile Inorganic Compounds (e.g., Hydrogen Sulfide)

2.2 Unless specified otherwise by the regulatory program, use a new, disposable, high capacity, 0.45 µm in-line filter.

2.3 Assemble the pump, tubing and filter as in 1.2 – 1.5 above.

2.4 Flush the filter as in 1.5.1 or 1.5.2 above.

2.5 Collect the samples as in 1.6 – 1.6.1.4 above.

Appendix FS 2200

Tables, Figures and Forms

Table FS 2200-1
Equipment for Collecting Groundwater Samples

Activity	Equipment Type
Well Purging	Variable speed centrifugal pump Variable speed submersible pump Variable speed bladder pump Variable speed peristaltic pump Bailer with lanyard: Not Recommended
Well Stabilization	pH meter DO meter Conductivity meter Thermometer/Thermistor Turbidimeter Flow-through cell Multi-function meters
Sample Collection	Variable speed peristaltic pump Variable speed submersible pump Variable speed bladder pump Bailer with lanyard (See Table FS 2200-3)
Filtration	Variable speed peristaltic pump Variable speed submersible pump Variable speed bladder pump Pressurized bailer 1.0 µm high capacity molded filter 0.45 µm high capacity molded filter
Groundwater Level	Electronic sensor Chalked tape

Table FS 2200-2
Dissolved Oxygen Saturation

TEMP	D.O.	mg/L	TEMP	D.O.	mg/L	TEMP	D.O.	mg/L	TEMP	D.O.	mg/L
deg C	SAT.	20%	deg C	SAT.	20%	deg C	SAT.	20%	deg C	SAT.	20%
15.0	10.084	2.017	19.0	9.276	1.855	23.0	8.578	1.716	27.0	7.968	1.594
15.1	10.062	2.012	19.1	9.258	1.852	23.1	8.562	1.712	27.1	7.954	1.591
15.2	10.040	2.008	19.2	9.239	1.848	23.2	8.546	1.709	27.2	7.940	1.588
15.3	10.019	2.004	19.3	9.220	1.844	23.3	8.530	1.706	27.3	7.926	1.585
15.4	9.997	1.999	19.4	9.202	1.840	23.4	8.514	1.703	27.4	7.912	1.582
15.5	9.976	1.995	19.5	9.184	1.837	23.5	8.498	1.700	27.5	7.898	1.580
15.6	9.955	1.991	19.6	9.165	1.833	23.6	8.482	1.696	27.6	7.884	1.577
15.7	9.934	1.987	19.7	9.147	1.829	23.7	8.466	1.693	27.7	7.870	1.574
15.8	9.912	1.982	19.8	9.129	1.826	23.8	8.450	1.690	27.8	7.856	1.571
15.9	9.891	1.978	19.9	9.111	1.822	23.9	8.434	1.687	27.9	7.842	1.568
16.0	9.870	1.974	20.0	9.092	1.818	24.0	8.418	1.684	28.0	7.828	1.566
16.1	9.849	1.970	20.1	9.074	1.815	24.1	8.403	1.681	28.1	7.814	1.563
16.2	9.829	1.966	20.2	9.056	1.811	24.2	8.387	1.677	28.2	7.800	1.560
16.3	9.808	1.962	20.3	9.039	1.808	24.3	8.371	1.674	28.3	7.786	1.557
16.4	9.787	1.957	20.4	9.021	1.804	24.4	8.356	1.671	28.4	7.773	1.555
16.5	9.767	1.953	20.5	9.003	1.801	24.5	8.340	1.668	28.5	7.759	1.552
16.6	9.746	1.949	20.6	8.985	1.797	24.6	8.325	1.665	28.6	7.745	1.549
16.7	9.726	1.945	20.7	8.968	1.794	24.7	8.309	1.662	28.7	7.732	1.546
16.8	9.705	1.941	20.8	8.950	1.790	24.8	8.294	1.659	28.8	7.718	1.544
16.9	9.685	1.937	20.9	8.932	1.786	24.9	8.279	1.656	28.9	7.705	1.541
17.0	9.665	1.933	21.0	8.915	1.783	25.0	8.263	1.653	29.0	7.691	1.538
17.1	9.645	1.929	21.1	8.898	1.780	25.1	8.248	1.650	29.1	7.678	1.536
17.2	9.625	1.925	21.2	8.880	1.776	25.2	8.233	1.647	29.2	7.664	1.533
17.3	9.605	1.921	21.3	8.863	1.773	25.3	8.218	1.644	29.3	7.651	1.530
17.4	9.585	1.917	21.4	8.846	1.769	25.4	8.203	1.641	29.4	7.638	1.528
17.5	9.565	1.913	21.5	8.829	1.766	25.5	8.188	1.638	29.5	7.625	1.525
17.6	9.545	1.909	21.6	8.812	1.762	25.6	8.173	1.635	29.6	7.611	1.522
17.7	9.526	1.905	21.7	8.794	1.759	25.7	8.158	1.632	29.7	7.598	1.520
17.8	9.506	1.901	21.8	8.777	1.755	25.8	8.143	1.629	29.8	7.585	1.517
17.9	9.486	1.897	21.9	8.761	1.752	25.9	8.128	1.626	29.9	7.572	1.514
18.0	9.467	1.893	22.0	8.744	1.749	26.0	8.114	1.623	30.0	7.559	1.512
18.1	9.448	1.890	22.1	8.727	1.745	26.1	8.099	1.620	30.1	7.546	1.509
18.2	9.428	1.886	22.2	8.710	1.742	26.2	8.084	1.617	30.2	7.533	1.507
18.3	9.409	1.882	22.3	8.693	1.739	26.3	8.070	1.614	30.3	7.520	1.504
18.4	9.390	1.878	22.4	8.677	1.735	26.4	8.055	1.611	30.4	7.507	1.501
18.5	9.371	1.874	22.5	8.660	1.732	26.5	8.040	1.608	30.5	7.494	1.499
18.6	9.352	1.870	22.6	8.644	1.729	26.6	8.026	1.605	30.6	7.481	1.496
18.7	9.333	1.867	22.7	8.627	1.725	26.7	8.012	1.602	30.7	7.468	1.494
18.8	9.314	1.863	22.8	8.611	1.722	26.8	7.997	1.599	30.8	7.456	1.491
18.9	9.295	1.859	22.9	8.595	1.719	26.9	7.983	1.597	30.9	7.443	1.489

Derived using the formula in Standard Methods for the Examination of Water and Wastewater, Page 4-101, 18th Edition, 1992

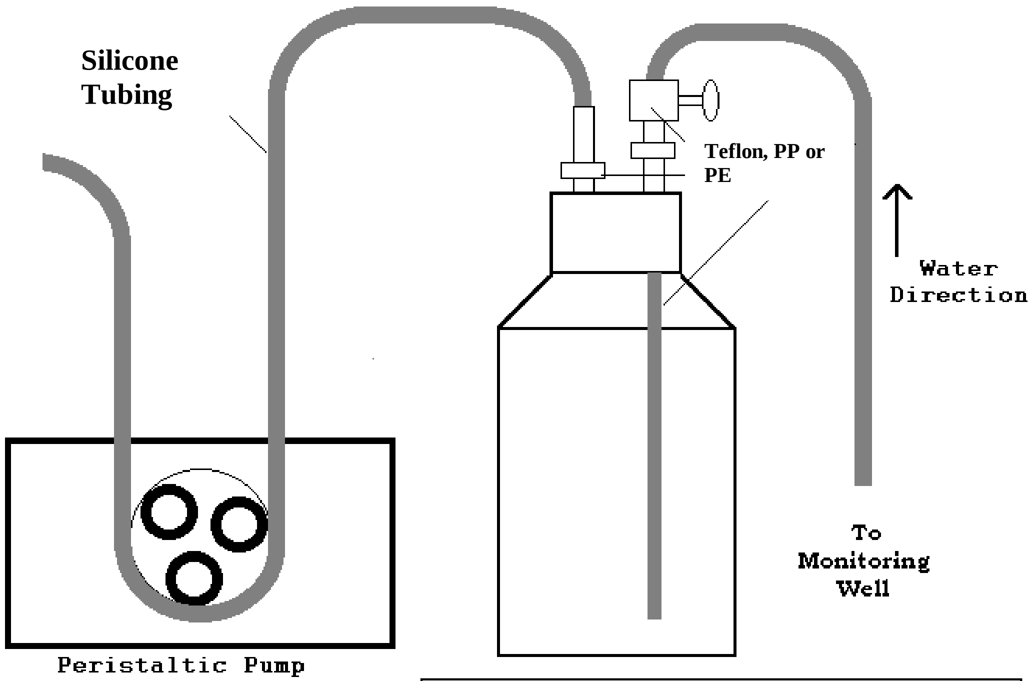
Table FS 2200-3
Allowable Uses for Bailers

• ANALYTE GROUP(S)	• PURGING (Not Recommended)	• SAMPLING	
	Use:	Use:	Not Recommended:
Volatile Organics Extractable Organics Radionuclides, including Radon Metals Volatile Sulfides	If allowed by permit, program, contract or order or If operated by a skilled individual with documented training in proper techniques. Field documentation must demonstrate that the procedure in FS 2213, section 4 was followed without deviation.	If concentrations exceed action levels, the purpose is to monitor effective treatment, and the DEP program allows the use of bailers; or If specified by DEP permit, program, contract or order. or If operated by a skilled individual with documented training in proper techniques and using appropriate equipment. Field documentation must demonstrate that the procedure in FS 2221, section 2 was followed without deviation.	If concentrations are near or below the stated action levels; or If a critical decision (e.g., clean closure) will be made based on the data; or If data are to demonstrate compliance with a permit or order.
Petroleum Hydrocarbons (TRPH) & Oil & Grease	If allowed by permit, program, contract or order or If operated by a skilled individual with documented training in proper techniques. Field documentation must demonstrate that the procedure in FS 2213, section 4 was followed without deviation.	Only if allowed by permit, program, contract or order as samples should be collected into the container without intermediate devices.	Unless allowed by permit, program, contract or order.

• **ANALYTE GROUP(S)**	• **PURGING** (Not Recommended)	• **SAMPLING**	
	Use:	Use:	Not Recommended:
Biologicals Inorganic Non-Metallics Aggregate Organics Microbiological Physical and Aggregate Properties	If allowed by permit, program, contract or order or If operated by a skilled individual with documented training in proper techniques. Field documentation must demonstrate that the procedure in FS 2213, section 4 was followed without deviation.	If all analytes collected from the well can be collected with a bailer; or If collected <u>after</u> collecting all analytes that require the use of a pump.	Before collecting any analytes that must be collected with a pump.
Ultra-Trace Metals	Never	Never	

Figure 2200-1

Pump and Trap for Extractable Organics



The glass sample bottle must be threaded to use a reusable sampling cap lined and installed with fittings made of Teflon, polypropylene or polyethylene, similar to the design shown.

Scenario 1: WELL SCREEN COMPLETELY SUBMERGED

Option 1a: Minimal Purge Volume: Pump or tubing is placed within the middle of the screen interval. The following conditions must be met to use this option:

1. The well screen interval is $\leq$ 10 feet.
2. Although drawdown may occur in the well when purging is initiated, the drawdown has to stabilize (Aquifer Recovery Rate = Purge Rate).
3. The samples will be obtained with the same equipment that was used to purge the well. Therefore, centrifugal pumps and bailers are not suitable for use in Option 1a.

If one or more of these conditions do not apply, use Option 1b.

Option 1b: Conventional Purge: Pump, tubing, or bailer [1] is placed above the screen at the top of the water column.

[1] DEP does not recommend the use of a bailer for purging; however, if a bailer is used it shall be lowered and raised at the rate of 2 cm/sec in the top of the water column.

Scenario 2: WELL SCREEN PARTIALLY SUBMERGED

Option 2a: A bailer [1] is placed at the top of the water column and is used to purge and sample the well.

Option 2b: Pump or tubing is placed within the middle of the saturated portion of the screen interval.

If the pump or tubing that was used for purging will not be used to obtain the sample, then position the pump or tubing at the top of the water column for purging.

Purging Procedure #1

1. After the drawdown in the well stabilizes, purge at least one equipment volume then collect the first set of stabilization parameters.
2. Thereafter, collect stabilization parameters $\geq$ 2 to 3 minutes apart.
3. Purge at least three equipment volumes before sampling.

Purging Procedure #2

1. Purge at least one well volume then collect first set of stabilization parameters.
2. Thereafter, collect stabilization parameters $\geq$ every 1/4 well volume.

Purging Procedure #3

1. Purge at least one well volume then collect first set of stabilization parameters.
2. Thereafter, collect stabilization parameters $\geq$ 2 to 3 minutes apart.

Purging Completion

If Dissolved Oxygen is $\leq$ 20% of saturation for the measured temperature and Turbidity is $\leq$ 20 NTUs, then purging is complete when three consecutive readings of the parameters listed below are within the following ranges:

Temperature $\pm$ 0.2 °C
pH $\pm$ 0.2 Standard Units
Specific Conductance $\pm$ 5.0% of reading

If Dissolved Oxygen (DO) is > 20% of saturation for the measured temperature and/or Turbidity is > 20 NTUs after every attempt has been made to reduce DO and/or turbidity, then purging is complete when three consecutive readings of the parameters listed below are within the following ranges:

Temperature $\pm$ 0.2 °C
pH $\pm$ 0.2 Standard Units
Specific Conductance $\pm$ 5.0% of reading
Dissolved Oxygen $\pm$ 0.2 mg/L or readings are within 10% (whichever is greater).
Turbidity $\pm$ 5 NTUs or readings are within 10% (whichever is greater).

If the well is expected to purge dry, position the pump or tubing within the screened interval and purge at $\leq$ 100 mL/minute until two equipment volumes are removed. Use the same pump for purging and sampling.

If the well purges dry at the lowest achievable flow rate (pumping at 100 mL/minute or less), then after a sufficient amount of water recharges in the well, collect the samples.

In either case listed above, before samples are collected, measure (once) pH, temperature, specific conductance, dissolved oxygen, and turbidity.

If one or more parameters do not stabilize after 5 volumes of the screened interval (purging procedure #1) or 5 well volumes (purging procedure #s 2 & 3) are removed, purging may be discontinued at the discretion of the sampling team leader.

FS 3000. SOIL

See also the following Standard Operating Procedures:

- FA 1000 Administrative Procedures
- FC 1000 Cleaning/Decontamination Procedures
- FD 1000 Documentation Procedures
- FM 1000 Field Planning and Mobilization
- FQ 1000 Field Quality Control Requirements
- FS 1000 General Sampling Procedures
- FT 1000 – FT 2000 Field Testing and Calibration

1. Introduction and Scope

1.1. Use these SOPs during field investigations to collect soil samples that are representative of current site conditions. It is very important to ensure that the collected samples are neither altered nor contaminated by sampling and handling techniques.

1.2. The following topics include: equipment choice, equipment construction materials, grab and areal or depth composite sampling techniques. Sample collection methods fall into three general depth classifications: surface, shallow subsurface, and deep subsurface. Once the samples are acquired, the handling procedures are very similar and are described below.

2. GENERAL

2.1. Select sampling equipment based on the type of sample to be collected and the analytes of interest. Choose soil sampling locations such that a representative portion of the soil is collected with minimal disturbance. Locations where natural vegetation is stressed or dead and/or areas that have surficial soil staining may be indicative of improper waste disposal practices.

2.2. If background and/or quality control sampling is warranted and feasible as determined in the site's work plan or by the project manager, select an up gradient, undisturbed location for obtaining the background and/or quality control samples. Be aware that differences in soil types may affect these background samples (e.g., sands vs. clays).

2.3. **Do not collect** samples for chemical analysis from auger flights or cuttings from hollow stem auger flights, except for waste characterization purposes for disposal.

2.4. Do not use samples that are collected for geological/lithological or vapor meter determinations for chemical analyses.

3. EQUIPMENT AND SUPPLIES

3.1. All equipment must be constructed of materials consistent with the analytes of interest. Refer to FS 1000, Tables FS 1000-1, FS 1000-2 and FS 1000-3 for selection of appropriate equipment and materials.

3.2. For information on sample container size and construction, see FS 1000, Table FS 1000-6.

3.3. For information on sampling equipment cleaning requirements, see FC 1000.

3.4. For information on preservation and holding time requirements, see FS 1000, Table FS 1000-6.

3.5. For information on documentation requirements, see FD 1000.

4. PROCEDURES FOR COMPOSITING

4.1. The following is not a complete discussion regarding all available sampling protocols nor the appropriateness or inappropriateness of compositing soil samples. The appropriateness of compositing soil samples will depend on the data quality objectives of the project. However, it is sometimes advantageous to composite soil samples to minimize the number of samples to be analyzed when sampling highly contaminated areas. Obtain permission from the DEP program.

4.1.1. Select sampling points from which to collect each aliquot.

4.1.2. Using the appropriate sampling technique, collect equal aliquots (same sample size) from each location and place in a properly cleaned container.

4.1.3. **Combine the aliquots of the sample directly in the sample container with no pre-mixing**.

4.1.4. Record the amount of each aliquot (volume or weight).

4.1.5. Label container, preserve on wet ice to 4°C and complete field notes.

4.1.6. Notify the laboratory that the sample is an unmixed composite sample, and request that the sample be thoroughly mixed before sample preparation or analysis.

5. SPECIFIC PROCEDURES FOR VOLATILE ORGANIC COMPOUNDS

Follow the procedures specified in EPA Method 5035 for sample collection and sample preparation. The protocols listed below **do not replace Method 5035** but clarify and/or modify certain method procedures. Therefore, it is essential that all organizations have a copy of Method 5035 as a reference document.

5.1. Container Preparation

5.1.1. All containers must be cleaned according to the FC 1000 sample container cleaning procedures for volatile organics.

5.1.2. Sample Vials: If sample vials are filled in the field, they must be provided with all reagents, stirring devices, label **and vial cap** to be used during sample analysis. These vials must be preweighed by the laboratory and records must be maintained so that there is an unambiguous link between the tare weight and the filled sample vial.

5.2. Collection Procedure

5.2.1. The sample vials (when used) will contain a premeasured amount of liquid. The laboratory must weigh the vials before sending into the field, and must weigh them again after receipt. Therefore:

- Do not lose any of the liquid either through evaporation or spillage
- Do not use a vial if some of the contents has spilled, or if it appears that some has leaked during transport
- Use the laboratory-supplied container label for identification information. **DO NOT apply any additional labels to the container**

- Do not interchange vial caps or septa

5.2.2. Minimize exposure to air by obtaining the sample directly from the sample source, using a coring device or a commercially designed sampling tool.

5.2.2.1. The sample collection device must be designed to fit tightly against the mouth of the vial or be small enough to be inserted into the vial. Use:

- EnCore or equivalent sampling devices or
- Disposable plastic syringes with the syringe end cut off prior to sampling (use **once** per sampling location).

5.2.2.2. Extrude the sample directly into the sample container.

5.2.3. Follow the method procedures for field transfer into the vial.

5.2.4. Procedures for determining the sample weight in the field are not required unless the project manager requires an accurate determination of the 5-gram sample size.

5.2.4.1. If the vials are returned to the laboratory for weighing, the sampler must be proficient in estimating the requisite 5-gram weight necessary for each sample.

5.2.4.2. If an accurate estimate of the 5-gram sample size is desired prior to starting sample collection activities, use a balance with a sensitivity of 0.1 gram. Check the balance calibration before each day's use with a set of weights that have been calibrated against NIST-traceable weights at least annually.

5.2.5. If the sampling device is transported to the laboratory with a sample, make sure the seals are intact, especially if collecting samples from sandy soils.

5.2.6. Collect at least two replicate samples from the same soil stratum and within close proximity to the original sample location.

5.2.7. Collect an additional aliquot of sample for screening and dry weight determinations.

5.3. <u>Preservation (see FS 1000, Table FS 1000-7)</u>

5.3.1. <u>Low Level (≤ 200 μg/kg volatile organics)</u>

5.3.1.1. Method 5035 discusses the use of sodium bisulfate, which is an acid. Since Florida soils contain significant amounts of calcium carbonate that reacts with acids, DEP does not recommend using this preservative.

5.3.1.2. Properly pack the samples (see FS 2004, section 5), and place all samples on wet ice.

5.3.1.3. Analyze unpreserved samples (no acid) within 48 hours.

5.3.1.4. Analyze acid-preserved samples within the specified 14-day holding time.

5.3.1.5. Analyze unpreserved samples that have been collected in a septum vial with premeasured analyte-free water within 48 hours.

5.3.1.6. If unpreserved samples collected in a septum vial with premeasured analyte-free water are frozen to -10°C at the laboratory within 48 hours of sample collection, analyze the samples within 14 days.

5.3.1.7. Analyze samples that have been collected with and transported in a sealed coring device within 48 hours.

5.3.1.8. If unpreserved samples collected in a sealed coring device are extruded from the corer into an appropriate liquid and frozen to -10°C at the laboratory within 48 hours of sample collection, analyze the samples within 14 days.

5.3.2. High Level (> 200 µg/kg volatile organics)

5.3.2.1. Properly pack the samples (see FS 2004, section 5), and place all samples on wet ice.

5.3.2.2. Analyze samples that have been collected with and transported in a sealed coring device within 48 hours.

5.3.2.3. If unpreserved samples collected in a sealed coring device are extruded from the corer into an appropriate liquid and stored at 4°C at the laboratory within 48 hours of sample collection, analyze the samples within 14 days.

5.3.2.4. Analyze samples that that have been preserved in methanol in the field within 14-days.

6. BULK SAMPLES: The collection of bulk samples will depend on the data quality objectives of the project.

6.1. Do not composite or mix VOC samples unless required by the DEP program or if mandated by a formal DEP document (permit, order or contract).

6.2. Select sampling points from which to collect each aliquot.

6.3. Using the appropriate sampling technique, collect equal aliquots (same sample size) from each location and place in a properly cleaned container.

6.3.1. **Combine the aliquots of the sample directly in the sample container with no pre-mixing**..

6.3.2. Pack soil tightly minimizing as much headspace as possible in the sample container.

6.3.3. Cap container tightly with Teflon side facing sample.

6.4. Record the amount of each aliquot (volume or weight) in the field notes.

6.5. Label container. Refer to FS 1000, Table FS 1000-7 for preservation and holding time requirements.

6.6. Notify the laboratory that the sample is an unmixed composite sample, and request that the sample be thoroughly mixed before sample preparation or analysis.

FS 3100. Surface Soil Sampling

Surface soil is generally classified as soil between the ground surface and 6-12 inches below ground surface.

1. Remove leaves, grass and surface debris from the area to be sampled.
2. Collect samples for volatile organic analyses as described in FS 3000, section 5.
3. Select an appropriate precleaned sampling device and collect the sample.
4. Transfer the sample to the appropriate sample container.
5. Clean the outside of the sample container to remove excess soil.

6. Label the sample container, place on wet ice to preserve to 4°C and complete the field notes.

FS 3200. Subsurface Soil Sampling

Interval begins at approximately 12 inches below ground surface.

FS 3210. SAMPLE COLLECTION PROCEDURE

Use the following after the desired depth has been reached by one of the methods outlined in FS 3220.

1. Collect samples for volatile organic analyses as described in FS 3000, section 5.
2. For other analyses, select an appropriate precleaned sampling device and collect the sample.
3. Transfer the sample to the appropriate sample container.
4. Clean the outside of the sample container to remove excess soil.
5. Label the sample container, place on wet ice to preserve to 4°C and complete the field notes.

FS 3220. REACHING THE APPROPRIATE DEPTH

1. SHOVELS AND DIGGERS: Used for soils from approximately 12 inches to a point when using the implement becomes impractical.

   1.1. Dig a hole or trench to the required depth.

   1.2. Follow the sample collection procedures outlined in FS 3210.

2. BACKHOE: Used for soils from approximately 12 inches to a point when using the implement becomes impractical.

   2.1. Dig a trench to the appropriate depth.

   2.2. Expose the sample, in the trench, by using a precleaned spoon, spatula or equivalent to clean away the soil that came in contact with the backhoe bucket.

   2.3. Use a **second** precleaned utensil to actually collect the sample from the trench.

   2.4. Follow the procedures outlined in FS 3210 to collect the sample.

3. BUCKET AUGERS AND HOLLOW CORERS: Suitable to reach soils from approximately 12 inches to a point when using the implement becomes impractical.

   3.1. Push and rotate the auger into the soil until the bucket is filled.

   3.2. Addition of a non-contaminating sleeve may allow an undisturbed soil sample to be obtained.

      3.2.1. The device consists of a standard auger head with a removable sleeve, which is inserted into the auger barrel. In this case it is the sleeve, which fills with soil.

      3.2.2. Remove the sleeve from the auger and cap.

   3.3. If the auger hole is prone to collapse due to low cohesion in some soils, DEP recommends inserting a temporary rigid PVC casing into the hole. The casing prevents hole collapse and minimizes cross-contamination between soil zones as the auger is advanced.

3.4. Remove the sample from the sampler by pushing or scraping the soil with an appropriate precleaned utensil into an appropriately precleaned tray or aluminum foil.

3.5. Remove any portion of the sample that has been disturbed and discard.

3.6. Follow the sample collection procedures outlined in FS 3210.

NOTE: If a confining layer has been breached during sampling, grout the hole to land surface with Type-1 Portland cement. This requirement may be different throughout Florida; contact the local Water Management District office for local requirements.

4. SPLIT SPOON SAMPLER: Suitable for reaching soils from approximately 12 inches to depths greater than 10 feet.

4.1. A split spoon sampler, useful for sampling unconsolidated soil, consists of two half cylinders (spoons) that fit together to form a tube approximately two feet in length and two inches in diameter.

4.1.1. The cylindrical arrangement is maintained by a retaining head and bit rings that screw on at each end of the split spoon.

4.1.2. The bit ring has beveled edges to facilitate sampling as the split spoon is forced into the ground.

4.1.3. Advance the sampler using the weight of the drilling stem and rods or a mechanical hammer.

4.1.4. Insert a catcher device in the head ring to prevent loss of unconsolidated sample during recovery.

4.2. After retrieving the split spoon sampler, expose the soil by unscrewing the bit and head rings and splitting the barrel.

4.3. If the recovery is enough to accommodate discarding a portion of the sample, discard the top and bottom two to three inches of the sample.

4.4. For volatile organic compounds collect the sample immediately from the **center portion of the split spoon** using the procedures described in FS 3000, section 5.

4.5. For other analyses, slice the sample from the center portion of the split spoon using a clean, decontaminated utensil.

4.6. Select an appropriate precleaned sampling device and collect the sample.

4.7. Transfer the sample to the appropriate sample container.

4.8. Clean the outside of the sample container to remove excess soil.

4.9. Label the sample container, place on wet ice to preserve to 4°C and complete the field notes.

5. DIRECT PUSH RIGS: May be used for depths greater than 10 feet below ground surface.

5.1. <u>Liners</u>: The clear liners are used with direct push rigs. This method is appropriate only for unconsolidated materials. The sampling depth that can be achieved varies depending on the rig and the lithologies that are encountered. Typically, the rig operator will:

- Place the liner inside the metal probe rod
- Select a point holder with an opening appropriate for the site lithology and screw it on the probe rod
- Advance the rod a full rod length
- Retrieve the rod
- Remove the point holder
- Remove the liner, and
- Slice the liner to expose the soil.

5.2. After the liner has been sliced, follow the procedures outlined in FS 3210, collecting volatile organic samples (if needed) immediately after the liner is sliced.

5.3. If samples for organic vapor analysis screening are required, collect them by slicing the sample(s) using a clean, decontaminated utensil and place them in 8-ounce (preferred) or 16-ounce jars, immediately cover the opening with aluminum foil and screw on the lid ring. If the contamination is derived from petroleum products, it is acceptable to use a clean gloved hand to transfer the sample(s) to the sample container(s).

5.4. For other analyses, slice the sample from the center portion of the split spoon using a clean, decontaminated utensil.

5.5. Select an appropriate precleaned sampling device and collect the sample.

5.6. Transfer the sample to the appropriate sample container.

5.7. Clean the outside of the sample container to remove excess soil.

5.8. Label the sample container, place on wet ice to preserve to 4°C and complete the field notes.

6. SHELBY TUBE SAMPLER

6.1. The Shelby tube sampler is used to sample unconsolidated soil and consists of a tube approximately 30 inches long and two inches (or larger) in diameter.

6.2. One end of the tube has edges beveled into a cutting edge. The other end can be mounted to an adapter, which allows attachment to the drilling rig assembly.

6.3. After drilling to the required depth with an auger or rotary drill bit, a soil sample is obtained through the auger or directly in the borehole.

6.4. Push the Shelby tube into the soil using the drilling rig's hydraulic ram or manually with a sledge hammer.

6.5. Remove the tube from the sampler head.

6.6. Extrude the sample from the Shelby tube.

6.7. Use a decontaminated utensil to remove any portion of the sample that has been disturbed.

6.8. Collect samples for volatile organics immediately from the center portion of the Shelby tube using the procedures described in FS 3000, section 5.

6.9. For other analyses, slice the sample from the center portion of the Shelby tube using a clean, decontaminated utensil.

6.10. Transfer the sample to the appropriate sample container.

6.11. Clean the outside of the sample container to remove excess soil.

6.12. Label the sample container, place on wet ice to preserve to 4°C and complete the field notes.

7. CORE BARREL

7.1. A standard core barrel is utilized when consolidated samples (such as limestone or dolomite) are to be sampled.

7.1.1. The core barrel is a cylinder approximately three feet long and two inches in diameter.

7.1.2. The barrel has a removable head ring with small embedded diamonds which allow the device to cut through rock or consolidated soil as the drilling rods are rotated.

7.2. Retrieve the sample core by unscrewing the head ring and sliding the sample into a precleaned container.

7.3. Use a decontaminated utensil to remove any portion of the sample that has been disturbed.

7.4. Remove the sample from the sampler (corer) with a precleaned tool.

7.5. Transfer the sample to the appropriate sample container.

7.6. Clean the outside of the sample container to remove excess soil.

7.7. Label the sample container, place on wet ice to preserve to 4°C and complete the field notes.

FT 1000. GENERAL FIELD TESTING AND MEASUREMENT

Use the following SOPs in conjunction with FT 1000:

- FD 1000 Documentation Procedures
- FM 1000 Field Planning and Mobilization
- FS 1000 General Sampling Procedures
- FT 1100 through FT 3000 Specific Field Testing Procedures

1. INTRODUCTION

1.1. Scope and Applicability: SOPs FT 1100 to FT 3000 outline procedures to conduct field testing measurements and observations. They include the parameters that are measured *in-situ* or in a field-collected sample. Additionally some samples with allowable extended holding times may be collected for laboratory measurement, as described in the specific FT-series SOPs. Included in SOPs FT 1100 to FT 3000 are:

- FT 1100 Field Measurement of Hydrogen Ion Activity (pH)
- FT 1200 Field Measurement of Specific Conductance (Conductivity)
- FT 1300 Field Measurement of Salinity
- FT 1400 Field Measurement of Temperature
- FT 1500 Field Measurement of Dissolved Oxygen (DO)
- FT 1600 Field Measurement of Turbidity
- FT 1700 Field Measurement of Light Penetration (Secchi Depth and Transparency)
- FT 1800 Field Measurement of Water Flow and Velocity
- FT 1900 Continuous Monitoring with Installed Meters
- FT 2000 Field Measurement of Residual Chlorine
- FT 3000 Aquatic Habitat Characterization

1.2. Exclusions: **If proposed for experimental purposes, field-screening procedures employing techniques not addressed in these SOPs** must be submitted to the DEP site or project manager. Such procedures must be addressed for each program or project dealing specifically with the planning and design of sampling events. Data quality objectives for quantitative assessment preclude the use of field-screening procedures for regulatory purposes.

1.3. Expectations and Requirements:

1.3.1. In some cases, specific instruments are identified in the SOP, with detailed instruction provided on their use. If you are using a different instrument from that identified in the SOP, follow the manufacturer's instructions for assembly, operation, and maintenance.

1.3.2. When required, the FT-series SOPs outline the instrument specifications. A field instrument must meet the stated requirements.

1.3.3. The FT-Series SOPs specify the calibration requirements for each method. Although instruments may vary in configuration or operation, the specified calibration requirements must be met.

1.3.3.1. Where applicable to the FT-series SOP, use the minimum number of calibration standards specified.

1.3.3.2. Do not establish the lower limit of the quantitative calibration bracket with “zero” solutions, quality control blanks or reagent dilution water.

1.3.4. Ensure that all equipment is in proper working condition, calibrated, and that batteries are properly charged before using the equipment for field testing measurements.

1.3.5. If reagents or standards are prepared from stock chemicals, they must be analytical reagent grade or better. Some procedures may specify a higher grade or assay of reagent or standard.

1.4. Recommendations for Use of Grab Samples or *in situ* Field Testing Measurements:

1.4.1. Use *in situ* readings where practical for field measurements in surface water and wastewater.

1.4.2. Use *in situ* readings or flow-through containers for field measurements for groundwater stabilization during purging and for other applications where groundwater monitoring measurements are required.

1.4.3. If grab samples are collected for measurement where allowed in the individual FT-series SOP, measure samples within fifteen (15) minutes of collection when immediate analysis is specified per Table FS 1000-4 and FS 1000-5. Otherwise, analyze grab samples within the applicable holding times specified in Table FS 1000-4 and FS 1000-5.

2. MINIMUM CALIBRATION REQUIREMENTS:

2.1. Calibration Definitions: This section outlines the essential calibration concepts that must be applied to each field test. Specific requirements for calibration are addressed in the individual SOPs.

2.1.1. Initial Calibration (IC): The instrument or meter electronics are adjusted (manually or automatically) to a theoretical value (e.g., dissolved oxygen saturation) or a known value of a calibration standard.

2.1.2. Initial Calibration Verification (ICV): The instrument or meter calibration is checked or verified directly following initial calibration by measuring a calibration standard of known value as if it were a sample and comparing the measured result to the calibration acceptance criteria listed in the SOP.

2.1.3. Continuing Calibration Verification (CCV): The instrument or meter calibration is checked or verified by measuring a calibration standard of known value as if it were a sample and comparing the measured result to the calibration acceptance criteria listed in the SOP.

2.1.4. Chronological Calibration Bracket: The interval of time between verifications within which environmental sample measurements must occur. The instrument or meter is calibrated or verified before and verified after the time of environmental sample measurement(s).

2.1.5. Quantitative Calibration Bracket: The instrument or meter is calibrated or verified at two known values that encompass the range of observed environmental sample measurement(s).

2.1.6. Acceptance Criteria: The numerical limits within which calibration verifications are acceptable.

2.2. Calibration Activities: Specific calibration procedures are given in the individual SOPs.

2.2.1. Chronological Calibration Bracket:

2.2.1.1. Ensure that the field test result is preceded by an acceptable ICV or CCV and followed by an acceptable CCV.

2.2.1.2. Specific requirements for chronological bracketing are addressed in the individual FT-series SOPs.

2.2.2. Quantitative Calibration Bracket:

2.2.2.1. Choose two standards that bracket the range of sample measurements. These standards may be used for initial calibrations or for verifications.

2.2.2.2. Specific requirements for quantitative bracketing are addressed in the individual FT-series SOPs.

2.2.3. Initial Calibration: Calibrate if no initial calibration has been performed or if a calibration verification does not meet acceptance criteria. Do not reuse standards for initial calibrations.

Table FT 1000-1: Field Testing Acceptance Criteria

Parameter	Acceptance Criteria
pH (FT 1100)	**± 0.2 Standard pH Units of buffer or more stringent program criteria**
Specific Conductance (FT 1200)	**± 5% of standard value**
Temperature (FT 1400)	**± 0.2^{o}C of NIST-traceable value (with correction factors) Verification over range of applicable values**
Dissolved Oxygen (FT 1500)	**± 0.3 mg/L of theoretical value (see Table FT 1500-1)**
Turbidity (FT 1600)	**0.1-10 NTU: ± 10% of standard value 11-40 NTU: ± 8% of standard value 41-100 NTU: ± 6.5% of standard value > 100 NTU: ± 5% of standard value**
Total Residual Chlorine (FT 2000)	**0.995 calibration curve correlation coefficient ± 10% of primary standard value ± 10% of secondary standard value Color comparator acceptance criterion: ± 10% of primary standard value**

2.2.4. Initial Calibration Verification:

2.2.4.1. Perform an ICV immediately after calibration. All ICVs must meet the calibration acceptance criteria specified in the applicable FT-series SOP. See Table FT 1000-1 for a list of acceptance criteria for the most common field testing procedures.

2.2.4.2. If an ICV fails to meet acceptance criteria, immediately recalibrate the instrument using the applicable initial calibration procedure or remove it from service.

2.2.5. Continuing Calibration Verification: Perform a CCV at no more than 24-hour intervals from previous verification, except where noted for individual FT-series SOPs.

2.2.5.1. If historically generated data demonstrate that a specific instrument remains stable for longer periods of time, the time interval between calibration verifications may be increased.

2.2.5.2. Base the selected time interval on the shortest interval that the instrument maintains stability. If CCVs consistently fail, shorten the time period between verifications or replace/repair the instrument.

2.2.5.3. All CCVs must meet the calibration acceptance criteria specified in the applicable FT-series SOP. See Table FT 1000-1 for a list of acceptance criteria for the most common field testing procedures.

2.2.5.4. If a CCV fails to meet acceptance criteria perform one or more of the following procedures as necessary:

- Reattempt the CCV again within the chronological bracket time interval without changing the instrument calibration. Do not perform maintenance, repair, or cleaning of the instrument or probe. Probes may be rinsed with analyte-free water or fresh verification standard. The CCV may be reattempted with a fresh aliquot of verification standard.
- Perform the initial calibration, perform an ICV, re-analyze the sample(s), and perform a CCV.
- Report all results between the last acceptable calibration verification and the failed calibration verification as estimated (report the value with a "J"). Include a narrative description of the problem in the field notes.

2.2.5.5. For installed instruments that are deployed for extended periods of time or used for continuous monitoring, see FT 1900.

2.2.5.6. Shorten the time period between verification checks or replace/repair the instrument.

2.2.6. Determining the Values of Secondary Standards: Use only those standards recommended by the manufacturer for a specific instrument. Only use secondary standards for continuing calibration verifications. See the individual FT-series SOPs for specific procedures for use of secondary standards. At documented intervals, determine or verify the values of secondary standards immediately after performing an initial calibration or after verifying the calibration with primary standards. Read each secondary standard as a sample. This result must be within the manufacturer's stated tolerance range and +/- 10% of the stated standard value. If the +/- 10% criterion is not met, assign this reading as the value of the standard. If the reading is outside the manufacturer's stated tolerance range, discard the secondary standard.

2.2.7. **More frequent calibration verifications may be required for discharge permit compliance measurements or other regulatory requirements.**

3. PREVENTIVE MAINTENANCE: Record all maintenance and repair notes in the maintenance logbook for each meter (see FS 1007). If rental equipment is used, a log is not required. However, the origin (i.e., rental company), rental date, equipment type, model number, and identification number (if applicable) must be entered into the field notes or a rental equipment notebook.

4. DOCUMENTATION

4.1. Standard and Reagent Documentation: Document information about standards and reagents used for calibrations, verifications, and sample measurements.

4.1.1. Note the date of receipt, the expiration date and the date of first use for all standards and reagents.

4.1.1.1. Document acceptable verification of any standard used after its expiration date.

4.1.2. Record the concentration or other value for the standard in the appropriate measurement units.

4.1.2.1. Note vendor catalog number and description for pre-formulated solutions as well as for neat liquids and powdered standards.

4.1.2.2. Retain vendor assay specifications for standards as part of the calibration record.

4.1.3. Record the grade of standard or reagent used.

4.1.4. When formulated in-house, document all calculations used to formulate calibration standards.

4.1.4.1. Record the date of preparation for all in-house formulations.

4.1.5. Describe or cite the procedure(s) used to prepare any standards in-house (DEP SOP or internal SOP).

4.2. <u>Field Instrument Calibration Documentation</u>: Document acceptable calibration and calibration verification for each instrument unit and field test or analysis, linking this record with affected sample measurements.

4.2.1. Retain vendor certifications of all factory-calibrated instrumentation.

4.2.2. Designate the identity of specific instrumentation in the documentation with a unique description or code for each instrument unit used.

4.2.2.1. Record the manufacturer name, model number, and identifying number such as a serial number for each instrument unit.

4.2.3. Record the time and date of all initial calibrations and all calibration verifications.

4.2.4. Record the instrument reading (value in appropriate measurement units) of all calibration verifications.

4.2.5. Record the name of the analyst(s) performing the calibration.

4.2.6. Document the specific standards used to calibrate or verify the instrument or field test with the following information:

- Type of standard or standard name (e.g., pH buffer)
- Value of standard, including correct units (e.g., pH = 7.0 SU)
- Manufacturer's tolerance range for secondary standards
- Link to information recorded according to section 4.1 above

4.2.7. Retain manufacturers' instrument specifications.

4.2.8. Document whether successful initial calibration occurred.

4.2.9. Document whether each calibration verification passed or failed.

4.2.10. Document any corrective actions taken to correct instrument performance according to records requirements of FD 3000.

4.2.10.1. Document the date and time of any corrective actions.

4.2.10.2. Note any incidence of discontinuation of use of the instrument due to calibration failure.

4.2.11. Describe or cite the specific calibration or verification procedure performed (DEP SOP or internal SOP).

4.3. Record all field-testing measurement data, to include the following:

- Project name
- Date and time of measurement or test (including time zone, if applicable)
- Source and location of the measurement or test sample (e.g., monitoring well identification number, outfall number, station number or other description)
- Latitude and longitude of sampling source location (if required)
- Analyte or parameter measured
- Measurement or test sample value
- Reporting units
- Initials or name of analyst performing the measurement
- Unique identification of the specific instrument unit(s) used for the test(s)

Appendix FT 1000
Tables, Figures and Forms

Table FT 1000-1 Field Testing Acceptance Criteria

Table FT 1000-1: Field Testing Acceptance Criteria	
Parameter	**Acceptance Criteria**
pH (FT 1100)	**+ 0.2 Standard pH Units of buffer or more stringent program criteria**
Specific Conductance (FT 1200)	**+ 5% of standard value**
Temperature (FT 1400)	**+ 0.2°C of NIST-traceable value (with correction factors) Verification over range of applicable values**
Dissolved Oxygen (FT 1500)	**+ 0.3 mg/L of theoretical value (see Table FT 1500-1)**
Turbidity (FT 1600)	**0.1-10 NTU: + 10% of standard value 11-40 NTU: + 8% of standard value 41-100 NTU: + 6.5% of standard value > 100 NTU: + 5% of standard value**
Total Residual Chlorine (FT 2000)	**0.995 calibration curve correlation coefficient + 10% of primary standard value + 10% of secondary standard value Color comparator acceptance criterion: + 10% of primary standard value**

FT 1100. Field Measurement of Hydrogen Ion Activity (pH)

Use in conjunction with:

- FT 1000 General Field Testing and Measurement
- FQ 1000 Field Quality Control Requirements
- FS 1000 General Sampling Procedures
- FD 1000 Documentation Procedures

1. Equipment and Supplies

1.1. Field Instrument: Use any pH meter consisting of a potentiometer, a glass electrode, a reference electrode, and a temperature-compensating device.

1.1.1. For routine fieldwork use a pH meter accurate and reproducible to at least 0.2-unit in the range of 0.0 to 14.0 units, and equipped with temperature-compensation adjustment. Record the pH value in pH units to one decimal place.

1.1.2. Advanced silicon chip pH sensors (with digital meters) may be used if demonstrated to yield equivalent performance to glass electrode sensors for the intended application.

1.2. Standards: Purchased or laboratory-prepared standard buffer solutions of pH values that bracket the expected sample pH range. Use buffers with nominal values of 4.0, 7.0 and 10.0 units for most situations. If the sample pH is outside the range of 4.0 to 10.0, then use two buffers that bracket the expected range with the pH 7 buffer being one of the two buffers. Alternatively, prepare appropriate standards per table I in method SM4500-H^+-B.

1.3. Recordkeeping and Documentation Supplies:

- Field notebook (w/ waterproof paper is recommended) or forms
- Indelible pens

2. Calibration and Use

2.1. General Concerns

2.1.1. The acceptance criterion for the initial calibration or the calibration verification is a reading of the standard within +/- 0.2-unit of the expected value.

2.1.2. On a weekly basis, check the calibration to ensure the % theoretical slope is greater than 90% (if applicable to your instrument type).

2.1.2.1. Note the % slope in the calibration records.

2.1.2.2. A % slope of less than 90% indicates a bad electrode that must be changed or repaired.

2.1.2.3. If % slope cannot be determined on your meter, or the manufacturer's optimum specifications are different, follow the manufacturer’s recommendation for maintaining optimum meter performance.

2.2. Interferences

2.2.1. Sodium at pH $\geq$ 10.0 units can be reduced or eliminated by using a low sodium error electrode.

2.2.2. Coatings of oils, greases, and particles may impair the electrode's response. Pat the electrode bulb dry with lint-free paper or cloth and rinse with de-ionized water. For cleaning hard-to-remove films, use acetone very sparingly so that the electronic surface is not damaged.

2.2.3. Temperature effects on the electrometric measurement of pH are controlled by using instruments having temperature compensation or by calibrating the meter at the temperature of the samples.

2.2.4. Poorly buffered solutions with low specific conductance (< 200 μmhos/cm) may cause fluctuations in the pH readings. Equilibrate electrode by immersing in several aliquots of sample before taking pH.

2.2.5. Ensure stable sample and sensor temperature before calibrating or taking sample readings. Drifting sensor or sample temperature may produce erroneous sample measurements, calibrations, or verifications.

2.2.6. Thoroughly rinse the pH sensor with deionized water or fresh buffer standard when calibrating or verifying the calibration or when taking sample measurements. For in-situ measurements, ensure adequate flushing of the sensor with fresh sample water prior to taking measurements. Any residual standard, sample or deionized water remaining on the sensor may affect the measurement of the subsequent standard or sample. This is especially true when samples or standards of widely different pH value are successively measured.

2.2.7. Drifting readings or an inability to calibrate the sensor may also indicate a fouled electrode. Clean the electrode per the manufacturer's instructions or replace.

2.3. Calibration: Follow the manufacturer's calibration instructions specific to your meter. Most instruments allow for a two-point calibration and a few models can perform a three-point calibration. Use the appropriate number of standard buffer solutions for calibration. Do not reuse buffers for initial calibrations.

2.3.1. Rinse the probe with de-ionized water (DI) before and between each standard buffer solution.

2.3.2. Follow the calibration activities specified in FT 1000, section 2.2.

2.3.2.1. Perform an initial calibration using at least two buffers. Always use a pH 7 buffer first.

2.3.2.2. If the pH sample range is expected to be wider than the range established by a two-point calibration (e.g., some samples at pH 4 and others at pH 8), then add a third calibration point. If the instrument cannot be calibrated with three buffers, the third buffer may be used as the initial calibration verification to extend the range.

2.3.2.3. After initial calibration, immediately perform an initial calibration verification (ICV). Read a buffer as a sample. To be acceptable, a calibration verification must be within +/- 0.2 pH units of the stated buffer value. For example, if reading the pH 4.0 buffer, the result must be in the 3.8 to 4.2 range. Certain regulatory programs may have more stringent acceptance criteria.

2.3.2.4. After sample measurement(s), perform a continuing calibration verification (CCV). Read a buffer as a sample. To be acceptable, a calibration verification must be within +/- 0.2 pH units of the stated buffer value. This CCV (if within acceptance criteria) can be used as the beginning of the chronological bracket. Certain regulatory programs may have more stringent acceptance criteria.

2.4. Measuring pH *in situ*: After calibrating the multi-probe sensors as outlined in 2.3 above, follow the meter's instructions to select the display for reading the pH of the sample. Immerse the probe at the desired depth in the water and wait for stabilization of the reading before recording the measurement.

2.5. Measuring pH in Flow-through Cells: When using a flow-through cell, the procedure described above in section 2.4 is applicable.

2.6. Measuring pH in Samples: After an acceptable initial calibration or calibration verification, follow these procedures to take a pH reading of a freshly collected sample (within 15 minutes of collection).

2.6.1. Pour enough of the fresh sample into a clean cup to take the reading.

2.6.2. Place the pH electrode in the sample (in the cup) and swirl the electrode.

2.6.3. Wait for stabilization, and read the pH value.

2.6.4. Turn the meter off after the last sample reading, rinse the electrode thoroughly with de-ionized water and replace the electrode's cap.

3. PREVENTIVE MAINTENANCE: Refer to FT 1000, section 3.

4. DOCUMENTATION

4.1. Standard and Reagent Documentation: Document information about standards and reagents used for calibrations, verifications, and sample measurements.

4.1.1. Note the date of receipt, the expiration date and the date of first use for all standards and reagents.

4.1.1.1. Document acceptable verification of any standard used after its expiration date.

4.1.2. Record the concentration or other value for the standard in the appropriate measurement units.

4.1.2.1. Note vendor catalog number and description for preformulated solutions as well as for neat liquids and powdered standards.

4.1.2.2. Retain vendor assay specifications for standards as part of the calibration record.

4.1.3. Record the grade of standard or reagent used.

4.1.4. When formulated in-house, document all calculations used to formulate calibration standards.

4.1.4.1. Record the date of preparation for all in-house formulations.

4.1.5. Describe or cite the procedure(s) used to prepare any standards in-house (DEP SOP or internal SOP).

4.2. Field Instrument Calibration Documentation: Document acceptable calibration and calibration verification for each instrument unit and field test or analysis, linking this record with affected sample measurements.

4.2.1. Retain vendor certifications of all factory-calibrated instrumentation.

4.2.2. Designate the identity of specific instrumentation in the documentation with a unique description or code for each instrument unit used.

4.2.2.1. Record manufacturer name, model number, and identifying number such as a serial number for each instrument unit.

4.2.3. Record the time and date of all initial calibrations and all calibration verifications.

4.2.4. Record the instrument reading (value in appropriate measurement units) of all calibration verifications.

4.2.5. Record the name of the analyst(s) performing the calibration.

4.2.6. Document the specific standards used to calibrate or verify the instrument or field test with the following information:

- Type of standard or standard name (e.g., pH buffer)
- Value of standard, including correct units (e.g., pH = 7.0 SU)
- Link to information recorded according to section 4.1 above

4.2.7. Retain manufacturers' instrument specifications.

4.2.8. Document whether successful initial calibration occurred.

4.2.9. Document whether each calibration verification passed or failed.

4.2.10. Document any corrective actions taken to correct instrument performance according to records requirements of FD 3000.

4.2.10.1. Document date and time of any corrective action.

4.2.10.2. Note any incidence of discontinuation of use of the instrument due to calibration failure.

4.2.11. Describe or cite the specific calibration or verification procedure performed (DEP SOP or internal SOP).

4.3. Record all field-testing measurement data, to include the following:

- Project name
- Date and time of measurement or test (including time zone, if applicable)
- Source and location of the measurement or test sample (e.g., monitoring well identification number, outfall number, station number or other description)
- Latitude and longitude of sampling source location (if required)
- Analyte or parameter measured
- Measurement or test sample value
- Reporting units
- Initials or name of analyst performing the measurement
- Unique identification of the specific instrument unit(s) used for the test(s)

FT 1200. Field Measurement of Specific Conductance (Conductivity)

Use in conjunction with:

- FT 1000 General Field Testing and Measurement
- FQ 1000 Field Quality Control Requirements
- FS 1000 General Sampling
- FD 1000 Documentation Procedures

1. INTRODUCTION: Specific conductance is a useful method to approximate the total amount of inorganic dissolved solids.

1.1. Conductivity varies with temperature. For example, the conductivity of salt water increases 3%/degree C at 0°C, and only 2%/degree C at 25°C.

1.2. Record the sample temperature or adjust the temperature of the samples prior to measuring specific conductance if the conductivity instrument does not employ automatic temperature compensation and correction of the instrument display value.

2. EQUIPMENT AND SUPPLIES

2.1. Field Instrument: Any self-contained conductivity instrument suitable for field work, accurate and reproducible to 5% or better over the operational range of the instrument, and preferably equipped with temperature-compensation adjustment. See references in FT 1210 below for additional information about instruments.

2.2. Standards: Purchased or laboratory-prepared standard potassium chloride (KCl) solutions with conductivity values that bracket the expected samples' range. In the laboratory, prepare standards of appropriate conductivities per SM2510 (Conductivity, in *Standard Methods for the Examination of Water and Wastewater, American Public Health Association*). Do not reuse standards for initial calibrations.

2.3. Recordkeeping and Documentation Supplies:

- Field notebook (w/ waterproof paper is recommended) or forms
- Indelible pens

3. CALIBRATION AND USE

3.1. General Concerns

3.1.1. Follow the instrument manufacturer's instructions for the details of operating the instrument.

3.1.2. For instruments without automatic temperature compensation, attempt to adjust the temperature of the samples to 25°C. If the temperature cannot be adjusted, measure the temperature with a calibrated device (see FT 1400), record the temperature, correct for temperature (per section 3.4 below) and report the results corrected to 25°C. See references in FT 1210 below for further information about temperature correction.

3.1.3. Ensure stable sample and sensor temperature before calibrating or taking sample readings. Drifting sensor or sample temperature may produce erroneous sample measurements, calibrations or verifications.

3.1.4. Thoroughly rinse the conductivity sensor with deionized water and fresh standard when calibrating or verifying the calibration or when taking sample measurements. For in-situ measurements, ensure adequate flushing of the sensor with fresh sample water prior to taking measurements. Any residual standard, sample or deionized water remaining on the sensor may affect the measurement of the subsequent standard or sample. This is especially true when samples or low-concentration standards are measured subsequent to measuring high-concentration standards.

3.1.5. Drifting readings or an inability to calibrate the sensor may also indicate a fouled electrode. Clean the electrodes per the manufacturer's instructions.

3.1.6. When successful calibration and verification cannot be achieved after ensuring that temperatures have stabilized and the sensor electrodes are clean and free of residual sample or standard from the previous measurement, suspect opened containers of standards, especially after repeated openings, when near the manufacturer's expiration date or when little standard volume remains in the container. Low-concentration conductivity standards are seldom stable for an extended period after opening.

3.2. Calibration and Calibration Verification:

3.2.1. Follow the calibration activities specified in FT 1000, section 2.2.

3.2.2. Initial Calibration: Calibrate the meter prior to use according to the following steps:

3.2.2.1. **Do not "zero" in the meter using analyte-free water or air.**

3.2.2.2. When the sample measurements are expected to be 100 μmhos/cm or greater, use two standard potassium chloride solutions that bracket the range of expected sample conductivities. A single standard at 100 μmhos/cm standard potassium chloride solution is acceptable for situations in which all sample measurements are expected to be less than 100 μmhos/cm.

3.2.2.3. Calibrate the instrument with one of the two standards to create an upper or lower boundary for the quantitative bracket.

3.2.2.4. Verify the calibration of the instrument with the second standard, quantitatively bracketing the range of expected sample values.

3.2.2.5. If the instrument can be calibrated with more than one standard, choose additional calibration standards within the range of expected sample values. The second standard in section 3.2.2.3 above may be used as an additional calibration standard.

3.2.2.6. Note: If all samples are expected to be less than 100 μmhos/cm, only one standard at 100 μmhos/cm standard potassium chloride solution is required.

3.2.3. Acceptability: Accept the calibration if the meter reads within +/- 5% of the value of any calibration standard used to verify the calibration. For example, the acceptance range for a 100 μmhos/cm standard is 95 to 105 μmhos/cm. If the meter does not read within +/- 5% of each calibration verification standard, determine the cause of the problem and correct before proceeding.

3.2.4. Temperature Correction: Most field instruments read conductivity directly. If the meter does not automatically correct values to 25°C, calculate correction factors using the procedure in section 3.4 below. Record all readings and calculations in the calibration records.

3.2.5. Continuing Calibration Verification: Check the meter in read mode with at least one KCl standard with a specific conductance which quantitatively brackets the conductivity measured in environmental samples. The reading for the calibration verification must also be within +/- 5% of the standard value (see 3.2.3 above).

3.2.5.1. If new environmental samples are encountered outside the range of the initial calibration in 3.2.2 above, verify the instrument calibration with an additional standard that brackets the range of new sample values. If these calibration verifications fail, recalibrate the instrument as in 3.2.2.

3.2.5.2. **More frequent calibration verifications may be required for discharge permit compliance measurements or other regulatory requirements.**

3.3. Measuring Specific Conductance of Samples:

3.3.1. Follow manufacturer's instructions for sample measurement.

3.3.2. Immerse or place the conductivity probe or sensor in situ at a measuring location representative of the sampling source.

3.3.3. Allow the conductivity instrument to stabilize.

3.3.4. Measure the water temperature (if necessary for manual temperature compensation) and record the temperature. See FT 1400 for temperature measurement procedures.

3.3.5. If the meter is equipped with manual temperature compensation, adjust the conductivity meter to the water temperature per manufacturer's instructions.

3.3.6. If the conductivity meter has a set of positions that multiply the reading by powers of ten in order to measure the full range of potential conductivities, set this dial to the correct range in order to take a reading.

3.3.7. Record the sample conductivity measurement reading within 15 minutes of water sample collection.

3.3.8. Rinse off the probe with de-ionized water. Follow manufacturer's instructions for probe storage between use.

3.4 Calculations for Temperature Compensation

If the meter does not automatically correct for temperature (manual or automatic adjustment), or if a probe with a cell constant other than 1 is used, the following formula must be used to normalize the data to 25°C:

$$K = \frac{(K_m)(C)}{1 + 0.0191(T-25)}$$

Where: K = conductivity in µmhos/cm at 25°C

K_m = measured conductivity in µmhos/cm at T degrees C

C = cell constant

T = measured temperature of the sample in degrees C

If the cell constant is 1, the formula for determining conductivity becomes:

$$K = \frac{(K_m)}{1 + 0.0191(T-25)}$$

Refer to SM2510B, 20th edition, if other calculations (i.e., determining cell constant, etc.) are required. See FT 1210 below.

3.5 *In situ* Measurements at Depth or With Flow-through Cells: After calibrating the instrument as outlined in 3.2 above, follow the manufacturer's instructions to measure the conductivity of the sample.

3.5.1. For *in situ* measurements immerse the probe at the desired depth and wait for stabilization of the reading and record its value. Follow a similar procedure when using a flow-through cell.

3.5.1.1 Preferably measure groundwater sample conductivity *in situ* with a downhole probe or in a flow-through system.

4. PREVENTATIVE MAINTENANCE: Refer to FT 1000, section 3.

5. DOCUMENTATION

5.1. Standard and Reagent Documentation: Document information about standards and reagents used for calibrations, verifications and sample measurements.

5.1.1. Note the date of receipt, the expiration date and the date of first use for all standards and reagents.

5.1.1.1. Document acceptable verification of any standard used after its expiration date.

5.1.2. Record the concentration or other value for the standard in the appropriate measurement units.

5.1.2.1. Note vendor catalog number and description for preformulated solutions as well as for neat liquids and powdered standards.

5.1.2.2. Retain vendor assay specifications for standards as part of the calibration record.

5.1.3. Record the grade of standard or reagent used.

5.1.4. When formulated in-house, document all calculations used to formulate calibration standards.

5.1.4.1. Record the date of preparation for all in-house formulations.

5.1.5. Describe or cite the procedure(s) used to prepare any standards in-house (DEP SOP or internal SOP).

5.2. Field Instrument Calibration Documentation: Document acceptable calibration and calibration verification for each instrument unit and field test or analysis, linking this record with affected sample measurements.

5.2.1. Retain vendor certifications of all factory-calibrated instrumentation.

5.2.2. Designate the identity of specific instrumentation in the documentation with a unique description or code for each instrument unit used.

5.2.2.1. Record manufacturer name, model number, and identifying number such as a serial number for each instrument unit.

5.2.3. Record the time and date of all initial calibrations and all calibration verifications.

5.2.4. Record the instrument reading (value in appropriate measurement units) of all calibration verifications.

5.2.5. Record the name of the analyst(s) performing the calibration.

5.2.6. Document the specific standards used to calibrate or verify the instrument or field test with the following information:

- Type of standard or standard name (e.g., conductivity standard)
- Value of standard, including correct units (e.g., conductivity = 100 µmhos/cm)
- Link to information recorded according to section 5.1 above

5.2.7. Retain manufacturers' instrument specifications.

5.2.8. Document whether successful initial calibration occurred.

5.2.9. Document whether each calibration verification passed or failed.

5.2.10. Document any corrective actions taken to correct instrument performance according to records requirements of FD 3000.

5.2.10.1. Document date and time of any corrective action.

5.2.10.2. Note any incidence of discontinuation of use of the instrument due to calibration failure.

5.2.11. Describe or cite the specific calibration or verification procedure performed (DEP SOP or internal SOP).

5.3. Record all field-testing measurement data, to include the following:

- Project name
- Date and time of measurement or test (including time zone, if applicable)
- Source and location of the measurement or test sample (e.g., monitoring well identification number, outfall number, station number or other description)
- Latitude and longitude of sampling source location (if required)
- Analyte or parameter measured
- Measurement or test sample value
- Reporting units
- Initials or name of analyst performing the measurement
- Unique identification of the specific instrument unit(s) used for the test(s)

FT 1300. Field Measurement of Salinity

Use in conjunction with:

- FT 1000 General Field Testing and Measurement
- FQ 1000 Field Quality Control Requirements
- FS 1000 General Sampling Procedures
- FD 1000 Documentation Procedures

1. INTRODUCTION: Salinity is an important property of industrial and natural waters. This field parameter is also important for assessing the source or origin of effluents and of the mixing between fresh and marine waters in coastal regions, in both surface water and groundwater.

1.1. Salinity is a unit-less parameter since by definition it is the ratio of the mass of dissolved salts to the total mass of a given volume of water. Thus, salinity values are commonly expressed as "grams of salt/kilograms of water" or $^{o}/_{oo}$.

1.2. Salinity is determined by using indirect methods involving the measurement of a related physical property such as conductivity, density, sound speed, or refractive index. The commonly used procedures in the field are determination of <u>conductivity</u> or <u>density</u> of the sample.

1.3. The sample salinity is calculated from an empirical relationship between salinity and the physical property as determined from a standard solution. Refer to the referenced method SM2520 for further discussions on these topics.

1.4. Because of its high sensitivity and easy of measurement, the conductivity method is most often used to determine the salinity. (Note – using a hydrometer to measure the density or the specific gravity to obtain an approximate salinity value is not recommended for reporting purposes.)

2. EQUIPMENT AND SUPPLIES

2.1. <u>Field Instrument</u>: Depending on the chosen method, use:

2.1.1. Any self-contained conductivity instrument with a platinum or graphite electrode type cell, and a temperature sensor. Some conductivity instruments have meter scales pre-calibrated for salinity and are sometimes referred to as Salinometers. For routine fieldwork use a conductivity meter accurate and reproducible to at least 5% or 1 μmho/cm (whichever is greater), and equipped with temperature-compensation adjustment; <u>or</u>

2.1.2. A precision "vibrating flow densimeter" (see Millero & Poisson, 1981) and a field thermometer.

2.2. <u>Standards</u>:

2.2.1. Purchased or laboratory-prepared Standard Seawater and/or potassium chloride (KCl) standards of appropriate equivalent salinities.

2.2.1.1. In the laboratory, prepare the Standard Seawater per recipe in method SM2520 and SM8010 (Table III), and standard KCl solutions per recipe in method SM2510 (American Public Health Association, American Water Works Association, Water Pollution Control Federation, <u>*Standard Methods for the Examination of Water and Wastewater)*</u>.

2.2.2. De-ionized water for calibration of the densimeter (if used).

2.3. Recordkeeping and Documentation Supplies:

- Field logbook (w/ waterproof paper is recommended) or field forms
- Indelible pens

3. CALIBRATION AND USE

3.1. Conductivity Method

3.1.1. Calibration: - Calibrate the instrument per manufacturer's instructions using one calibration standard, either standard seawater or a KCl solution, as applicable. The acceptance criterion for initial calibration or a calibration verification is that the instrument reading is within +/- 5% of the standard value. For example, when calibrating with standard seawater, S = 35, the meter must read in the 34 to 36 range in order to be acceptable.

3.1.1.1. Use standard seawater (S = 35) when measuring salinity in the open ocean or estuaries with a predominance of seawater.

3.1.1.2. KCl may be used in estuarine waters with low salinity (S = 0 – 40).

3.1.1.3. If verifying or calibrating with a "zero" standard, do not use analyte-free water or air check (dry electrode) as the blank.

3.1.1.4. If the meter does not provide a direct reading of salinity, use the equation found in SM2520B to convert the readings to salinity.

3.1.1.5. Follow the calibration activities in FT 1000, section 2.2.

3.1.1.6. Do not reuse standards for initial calibrations.

3.1.2. Field Use: - Rinse the probe with DI water after calibration and before each sample measurements. Follow the manufacturer's instructions for temperature compensation, if needed. Report salinities with only one decimal figure.

3.1.3. General Concerns for Conductivity Method

3.1.3.1. Ensure stable sample and sensor temperature before calibrating or taking sample readings. Drifting sensor or sample temperature may produce erroneous sample measurements, calibrations, or verifications.

3.1.3.2. Thoroughly rinse the conductivity (salinity) sensor with deionized water and fresh standard when calibrating or verifying the calibration or when taking sample measurements. For in-situ measurements, ensure adequate flushing of the sensor with fresh sample water prior to taking measurements. Any residual standard, sample, or deionized water remaining on the sensor may affect the measurement of the subsequent standard or sample. This is especially true when samples or low-concentration standards are measured subsequent to measuring high-concentration standards.

3.1.3.3. Drifting readings or an inability to calibrate the sensor may also indicate a fouled electrode. Clean the electrodes per the manufacturer's instructions.

3.1.3.4. When successful calibration and verification cannot be achieved after ensuring that temperatures have stabilized and the sensor electrodes are clean and free of residual sample or standard from the previous measurement, suspect opened containers of standards, especially after repeated openings, when near the manufacturer's expiration date or when little standard volume remains in the container. Low-concentration conductivity standards are seldom stable for an extended period after opening.

3.2. Density Method

The vibrating flow densimeter is an instrument that allows for precise and rapid measurements of the density of a liquid, such as water. The principle of operation is the effect of the density of the sample on the frequency of a vibrating tube encased in a constant-temperature jacket. The measurement is made by passing the water (sample) through the vibrating tube and reading the period of vibration that is electronically sensed and displayed by the densimeter. The sample density (D) is proportional to the square of the period of vibration (T):

$$D = a + bT^2$$

Where a and b are terms determined by calibration, b being determined by calibration of the densimeter with Standard Seawater. The difference between the density of the sample (D) and that of pure water (D_0) is given by:

$$D - D_0 = b\,(T^2 - T_0^2)$$

Where T and T_0 are, respectively, the periods of the sample and that of pure (de-ionized) water. Using this second equation, you only have to deal with the term b for calibration purposes. Hence, the system can be calibrated with two liquids: pure water and Standard Seawater. Follow the manufacturer's instruction for calibration of the densimeter.

The salinity of the sample is determined by the one-atmosphere international equation of state for seawater. This equation relates the difference ($D - D_0$) to the practical salinity as a function of the temperature of the sample (which is also measured by the densimeter or the field thermometer). For further details on this calculation read the referenced method SM2520C.

4. PREVENTIVE MAINTENANCE: Refer to FT 1000, section 3.

5. DOCUMENTATION

5.1. Standard and Reagent Documentation: Document information about standards and reagents used for calibrations, verifications, and sample measurements.

5.1.1. Note the date of receipt, the expiration date and the date of first use for all standards and reagents.

5.1.1.1. Document acceptable verification of any standard used after its expiration date.

5.1.2. Record the concentration or other value for the standard in the appropriate measurement units.

5.1.2.1. Note vendor catalog number and description for preformulated solutions as well as for neat liquids and powdered standards.

5.1.2.2. Retain vendor assay specifications for standards as part of the calibration record.

5.1.3. Record the grade of standard or reagent used.

5.1.4. When formulated in-house, document all calculations used to formulate calibration standards.

5.1.4.1. Record the date of preparation for all in-house formulations.

5.1.5. Describe or cite the procedure(s) used to prepare any standards in-house (DEP SOP or internal SOP).

5.2. Field Instrument Calibration Documentation: Document acceptable calibration and calibration verification for each instrument unit and field test or analysis, linking this record with affected sample measurements.

5.2.1. Retain vendor certifications of all factory-calibrated instrumentation.

5.2.2. Designate the identity of specific instrumentation in the documentation with a unique description or code for each instrument unit used.

5.2.2.1. Record manufacturer name, model number, and identifying number such as a serial number for each instrument unit.

5.2.3. Record the time and date of all initial calibrations and all calibration verifications.

5.2.4. Record the instrument reading (value in appropriate measurement units) of all calibration verifications.

5.2.5. Record the name of the analyst(s) performing the calibration.

5.2.6. Document the specific standards used to calibrate or verify the instrument or field test with the following information:

- Type of standard or standard name (e.g., salinity standard)
- Value of standard, including correct units (e.g., salinity = 20 $^{o}/_{oo}$)
- Link to information recorded according to section 5.1 above

5.2.7. Retain manufacturers' instrument specifications.

5.2.8. Document whether successful initial calibration occurred.

5.2.9. Document whether each calibration verification passed or failed.

5.2.10. Document any corrective actions taken to correct instrument performance according to records requirements of FD 3000.

5.2.10.1. Document date and time of any corrective action.

5.2.10.2. Note any incidence of discontinuation of use of the instrument due to calibration failure.

5.2.11. Describe or cite the specific calibration or verification procedure performed (DEP SOP or internal SOP).

5.3. Record all field-testing measurement data, to include the following:

- Project name
- Date and time of measurement or test (including time zone, if applicable)
- Source and location of the measurement or test sample (e.g., monitoring well identification number, outfall number, station number or other description)
- Latitude and longitude of sampling source location (if required)

- Analyte or parameter measured
- Measurement or test sample value
- Reporting units
- Initials or name of analyst performing the measurement
- Unique identification of the specific instrument unit(s) used for the test(s)

FT 1400. Field Measurement of Temperature

The use of this SOP is not required when using field temperature measurement devices to monitor groundwater stabilization during the purging of groundwater monitoring wells. Field temperature measurement devices used for temperature compensation (correction) for other measurements such as dissolved oxygen, specific conductance or pH are also exempted from the requirements of this SOP. FT 1400 must be used for all other field temperature measurements required by DEP.

Use this SOP in conjunction with the following DEP SOPs:

- FT 1000 General Field Testing and Measurement
- FQ 1000 Field Quality Control Requirements
- FS 1000 General Sampling Procedures
- FD 1000 Documentation Procedures

1. EQUIPMENT AND SUPPLIES

1.1. Field Instruments: Use any of the following instrument types for performing field measurements:

- Digital thermistor (thermocouple type) and meter typical of field instruments
- Glass bulb, mercury-filled thermometer (not recommended for field ruggedness)
- Glass bulb, alcohol-filled thermometer with protective case
- Bi-metal strip/dial-type thermometer
- Advanced silicon chip temperature sensor and digital meter

1.1.1. Field instruments must be capable of measuring temperature in 0.1^{o}C increments.

1.2. Standard Thermometer: NIST-traceable Celsius certified thermometer with scale marks for every 0.1^{o}C increment, a range of 0^{o}C to 100^{o}C (or a range bracketing expected sample temperatures) and correction chart supplied with certification. The standard thermometer must have a valid certification for the period of measurement.

1.3. Recordkeeping and Documentation Supplies:

- Field notebook or forms \
- Indelible pens

2. CALIBRATION AND USE

2.1. General Concerns

2.1.1. Select a temperature measuring device meeting the requirements of section 1.1 above.

2.1.2. Dial-type and thermocouple-type devices with meters are preferred over the glass thermometers for fieldwork because of their durability and ease of reading.

2.1.2.1. Transport glass thermometers in protective cases.

2.1.2.2. Inspect glass thermometers for liquid separation. Do not use a thermometer if the liquid has separated.

2.1.2.3. Most instruments with digital display will provide more decimal figures than are significant. Record the temperature reading with only one rounded decimal figure (e.g., 25.9 instead of 25.86°C).

2.2. Calibration

2.2.1. Follow the calibration activities specified in FT 1000, section 2.2.

2.2.2. Verify all thermistor (meter) devices and field thermometers against the NIST-traceable standard thermometer at several temperatures in the expected sample measurement range, using any correction factor indicated by the certificate supplied with the NIST-traceable thermometer.

2.2.2.1. See the US Geological Survey, National Field Manual for the Collection of Water-Quality Data, Book 9, Chapter A6, Field Measurements, Section 6.1, Temperature, Techniques of Water-Resources Investigations, 4/98 for additional guidance about making temperature comparisons with the standard thermometer.

2.2.2.2. Make note of the calibration in the calibration records. See section 4 below.

2.2.2.3. The field measurement device may be used with a linear correction factor provided that the observed temperature difference with the standard thermometer is documented at incremental temperatures over the range of expected sample temperatures.

2.2.2.4. Use the resulting correction factor when making temperature measurements of samples with the field measurement device.

2.2.2.5. Prominently display the correction factor on the field measurement device, with the date last verified. A calibration correction curve or plot may also be used.

2.2.2.6 To be acceptable, a calibration verification must be within +/- 0.5°C of the corrected reading of the NIST-traceable thermometer.

2.2.2.7 Properly dispose of glass-bulb thermometers that do not meet the above calibration acceptance criteria.

2.2.3. Continuing Calibration Verifications:

2.2.3.1. Determine the maximum time between continuing calibration verifications for the specific field temperature measurement device based on instrument stability.

2.2.3.2. Verify the field measurement device against the standard NIST-traceable thermometer as in section 2.2.2 above.

2.2.4. Refer to additional calibration requirements in FT 1000, section 2.2.

2.2.5. **More frequent calibration verifications may be required for discharge permit compliance measurements or other regulatory requirements.**

2.3. Measuring Sample Temperature

2.3.1. Insert or place the thermometer or sensor *in situ* at a measuring location representative of the sampling source.

2.3.2. Allow the thermometer or temperature sensor to equilibrate to ambient *in situ* temperature.

2.3.2.1. Groundwater samples must be measured *in situ* with a downhole probe or in a flow-through container. Do not measure bailed or pumped samples in an intermediate container containing static sample.

2.3.3. Record the temperature to the nearest 0.1°C after the reading stabilizes and remains constant.

3. PREVENTIVE MAINTENANCE: Refer to FT 1000, section 3.

4. DOCUMENTATION

4.1. Standards Documentation: Document information about the NIST-traceable standard thermometer in the calibration record, including:

- Unique identification for the thermometer
- Vendor certificate of calibration, including any correction factor
- Vendor's expiration date for the certificate of calibration

4.2. Field Instrument Calibration Documentation: Document acceptable calibration and calibration verification for each instrument unit and field test or analysis, linking this record with affected sample measurements.

4.2.1. Retain vendor certifications of all factory-calibrated instrumentation.

4.2.2. Designate the identity of specific instrumentation in the documentation with a unique description or code for each instrument unit used.

4.2.2.1. Record manufacturer name, model number, and identifying number such as a serial number for each instrument unit.

4.2.3. Record the time and date of all initial calibrations and all calibration verifications.

4.2.4. Record the instrument reading (value in appropriate measurement units) of all calibration verifications.

4.2.5. Record the name of the analyst(s) performing the calibration.

4.2.6. Document the following information about initial calibration and calibration verifications and link to information recorded according to section 4.1 above:

- Details of the method used to compare the field measurement device to the NIST-traceable standard thermometer.
- Results of each calibration verification, including the expected reading (per the NIST-traceable standard thermometer)
- The actual reading of the field measurement device, using any established correction factors and correct units.

4.2.7. Retain manufacturers' instrument specifications.

4.2.8. Document whether successful initial calibration occurred.

4.2.9. Document whether each calibration verification passed or failed.

4.2.10. Document any corrective actions taken to correct instrument performance (such as a new correction factor) according to records requirements of FD 3000.

4.2.10.1. Document date and time of any corrective action.

4.2.10.2. Note any incidence of discontinuation of use of the instrument due to calibration failure.

4.2.11. Describe or cite the specific calibration or verification procedure performed (DEP SOP or internal SOP).

4.3. Record all field-testing measurement data, to include the following:

- Project name
- Date and time of measurement or test (including time zone, if applicable)
- Source and location of the measurement or test sample (e.g., monitoring well identification number, outfall number, station number or other description)
- Latitude and longitude of sampling source location (if required)
- Analyte or parameter measured
- Measurement or test sample value
- Reporting units
- Initials or name of analyst performing the measurement
- Unique identification of the specific instrument unit(s) used for the test(s)

FT 1500. Field Measurement of Dissolved Oxygen (DO)

Use in conjunction with:

- FT 1000 General Field Testing and Measurement
- FS 1000 General Sampling Procedures
- FD 1000 Documentation Procedures

1. EQUIPMENT AND SUPPLIES

1.1. Field Instruments

1.1.1. Membrane-type polarographic or galvanic electrode DO sensor with dedicated meter or configured with multi-parameter sonde

1.1.2. Luminescence-based DO sensor with dedicated meter or configured with multi-parameter sonde (see American Society for Testing and Materials, *Standard Test Methods for Dissolved Oxygen in Water*, Test Method C-Luminescence-based Sensor, D 888-05).

1.1.3. Select instrument assemblies that provide minimum precision of +/- 0.2 mg DO/L and a minimum accuracy of +/- 0.2 mg DO/L.

1.1.4. Compensate for temperature dependence of DO measurements by using instruments employing automatic temperature compensation or by manually correcting measurements in accordance with SM 4500-O G (see *Standard Methods for the Examination of Water and Wastewater*, American Public Health Association, American Water Works Association, Water Pollution Control Federation).

1.1.4.1. Calibrate on-board temperature sensors as described in FT 1400.

1.2. Standards

1.2.1. NIST-traceable Celsius thermometer with a scale marked for every 0.1^{o}C and a range of 0 to 100^{o}C.

1.2.2. Access to an organization with capability to perform the Winkler titration procedure is recommended but not mandatory.

1.2.3. A "zero-DO standard", prepared on-site with an aliquot of the sample water, is optional. Prepare by adding excess sodium sulfite and a trace of cobalt chloride to bring the DO to zero.

1.3. Recordkeeping and Documentation Supplies:

- Field notebook (w/ waterproof paper is recommended) or forms
- Indelible pens

2. CALIBRATION AND USE: the electrode method is predominantly used *in-situ* for dissolved oxygen determinations.

2.1. General Concerns

2.1.1. Turbulence is necessary to keep a constant flow of water across the membrane-sample interface. Make sure the appropriate mechanism is working before using the probe.

2.1.2. Follow instrument manufacturer's instructions for probe storage. For example, store the probe with a cover that creates a saturated atmosphere. A cap, with a wet sponge in it, will suffice for single-parameter probes. If the sensor is in a multi-probe device, keep the protective cap chamber moist during storage.

2.1.3. Before mobilizing, check to make sure there are no bubbles beneath the probe membrane, or any wrinkles or tears in the probe membrane. If so, replace the membrane and KCL solution. Check the leads, contacts, etc. for corrosion and/or shorts if meter pointer remains off-scale, does not calibrate, or drifts.

2.1.4. Dissolved inorganic salts interfere with the performance of DO probes. For example, DO readings in salt water are affected by the salinity and must be corrected. The DO meter may adjust automatically based on readings taken from the specific conductivity/salinity probe. If corrections are not automatic the appropriate calculations must be used to correct for salinity. If automatic adjustments are used the specific conductivity/salinity probe calibration must be verified or calibrated in accordance with FT1200.

2.1.5. Reactive gases, which pass through the membrane, may interfere. For example, chlorine will depolarize the cathode and cause a high probe output. Long-term exposures to chlorine will coat the anode with the chloride of the anode metal and eventually desensitize the probe. Sulfide (from H_2S) will undergo oxidation if high enough potential (voltage) is applied, creating current flow, yielding faulty readings. If such interferences are suspected, change the membrane electrode more frequently and calibrate at more frequent intervals.

2.1.6. Ensure that the temperature of the sensor and sample are stable. Unstable temperatures will produce erroneous calibrations, verifications or sample measurements.

2.1.7. Erroneous calibrations or verifications may result if the saturated air chamber is not vented to atmospheric pressure, properly humidified and protected from temperature fluctuations produced by common field conditions such as evaporation or fluctuation in sunlight intensity.

2.2. Follow the quality control requirements for calibration (see activities in FT 1000, section 2.2).

2.3. Initial Calibration and Initial Calibration Verification

2.3.1. Air Calibration and Initial Calibration Verification (ICV): Calibrate the meter at 100% saturation. Before use, verify the meter calibration in water-saturated air to make sure it is properly calibrated and operating correctly. Make a similar verification at the end of the day or sampling event. Follow the manufacturer's instructions for your specific instrument.

2.3.1.1. Allow an appropriate warm up period before initial field calibration.

2.3.1.2. Wet the inside of the calibration chamber with water, pour out the excess water (leave a few drops), wipe any droplets off the membrane/sensor and insert the sensor into the chamber (this ensures 100% humidity).

2.3.1.3. Allow adequate time for the DO sensor and the air inside the calibration chamber to equilibrate.

2.3.1.4. Once the probe/calibration chamber is stable at ambient temperature, check the air temperature and determine, from the DO versus temperature table, what the DO saturation value should be at the observed temperature (see Table FT

1500-1, below). A stable and accurate temperature is required for a valid calibration. The acceptance criterion for DO calibration verification is +/- 0.3 mg DO/L at the observed temperature of the verification.

2.4. Continuous Calibration Verification

2.4.1. Air-Calibration Verification: DO sensor or instrument is calibrated against air that is saturated with water at a known temperature and ambient atmospheric pressure. Use Table FT 1500-1 below to verify calibration at specified temperature.

2.4.1.1. Wet the inside of the calibration chamber with water, pour out the excess water (leave a few drops) and insert the sensor into the chamber (this ensures 100-percent humidity)

2.4.1.2. Allow adequate time for the DO sensor and the air inside the calibration chamber to equilibrate.

2.4.1.3. Measure the temperature in the calibration chamber and observe the readings until the instrument stabilizes.

2.4.1.4. Use the oxygen solubility Table FT 1500-1 below to determine the DO saturation at a measured temperature and atmospheric pressure. Calculate values to the nearest tenth degree by interpolation or use an expanded version of this table found in FS 2200, which provides saturation data in 0.1 ^{o}C increments for a selected temperature range (see Table FS 2200-2).

2.4.1.5. Compare DO meter reading with value obtained from Table FT 1500-1 below to verify continuous calibration.

2.5. Additional Verifications: The following methods may be used as additional checks to verify calibration. These additional checks may be required as part of a specific permit.

2.5.1. Winkler method: This check is useful to assess the condition of the DO sensor (i.e., its degradation with time/use) and that the instrument can still maintain a valid calibration (see SM 4500-O C).

2.5.1.1. **Perform the Winkler method when required by permit or other regulation at the required calendar frequency.**

2.5.1.2. For an accuracy calibration verification using the Winkler method, follow SM 4500-O C.

2.5.1.3. Fill a clean bucket with uncontaminated or de-ionized water and place the probe into the bucket (with stirrer or equivalent mechanism turned off). Fill at least two biological oxygen demand (BOD) bottles without entraining atmospheric oxygen into the bottles. Carefully submerge the bottom of the bottle (one at a time) into the water and allow the water to fill the bottle. Place the bottle on the bottom of the bucket and carefully place stopper into it without adding atmospheric oxygen. Retrieve the bottles and determine their DO by the Winkler method (see SM4500-O-C for more details). Turn the stirrer or equivalent mechanism on and read the DO of the water in the bucket.

2.5.1.4. Adjust the DO meter according to manufacturer's instructions. Be sure to adjust the meter to the temperature of water in the bucket, and then calibrate the DO meter to read the average DO concentration of the two samples determined by the Winkler test.

2.5.2. <u>Zero-DO Verification</u>: The air calibration and the interfering effects of the sample can be further checked in the field by means of a "zero-DO standard"(SM 4500-O G).

2.5.2.1. Prepare this standard on-site with an aliquot of the sample by adding excess sodium sulfite and a trace of cobalt chloride to bring the DO to zero. Prepare this zero-DO standard in a beaker or a large-mouth sample container of appropriate size to insert the DO probe.

2.5.2.2. After adding the chemicals, gently swirl the water and let it sit for about 30 seconds before inserting the probe.

2.5.2.3. Read the DO of the sample. If the reading is outside the acceptance interval, the instrument must be recalibrated and/or zero-adjusted if the meter allows for this adjustment.

2.5.3. <u>Air-Saturated Water</u>: The DO sensor or instrument system is calibrated against water that is saturated with oxygen at a known temperature and ambient atmospheric pressure.

2.5.3.1. The temperature and conductivity of water used for calibration should be about the same as the temperature and conductivity of the water to be measured.

2.5.3.2. Place DO sensor and calibration water in a large beaker or open-mouth container.

2.5.3.3. Aerate the water for an adequate amount of time.

2.5.3.4. Determine if the water is 100 percent saturated with oxygen, and take a temperature reading. Temperature must be calibrated or verified for accuracy before DO calibration verification.

2.5.3.5. Use Table FT 1500-1 above to determine the DO saturation value at the measured water temperature. Compare DO meter reading with value obtained from Table FT 1500-1 to ensure continuous calibration.

2.6. <u>Measuring DO in Samples</u>:

2.6.1. Insert or place the DO probe *in situ* at a measuring location representative of the sampling source:

2.6.1.1. Take the DO of an effluent just before it enters the receiving water. If the effluent aerated prior to entering the surface water, take the DO reading in the receiving water right where it enters.

2.6.1.2. For well mixed surface waters, e.g., fast flowing streams, take the DO reading at approximately 1-2 feet below the surface or at mid-depth.

2.6.1.3. For still or sluggish surface waters, take a reading at one foot below the surface, one foot above the bottom, and at mid-depth.

2.6.1.4. If it is shallow surface waters, (less than two feet) take the reading at mid-depth.

2.6.1.5. Do not take a reading in frothy or aerated water unless required by the sampling plan.

2.6.1.6. Groundwater samples must be measured *in situ* with a downhole probe or in a flow-through container. Do not measure bailed or pumped samples in an intermediate container containing static sample.

2.6.2. Rinse probe with de-ionized water and keep the probe in the saturated atmosphere (see 2.1.2 above) between sites and events.

2.6.3. If the readings show distinct, unexplainable changes in DO levels, or when the probe has been in waters with high sulfides, recalibrate or perform maintenance per manufacturer's instructions. While taking a reading, if it is very low (e.g., below 1.0 mg/L), allow the meter to stabilize, record it and then, remove and rinse the probe, as the environment is very likely anoxic and may contain hydrogen sulfide, which can damage the probe.

2.6.4. Salinity and Temperature corrections may be necessary. Follow manufacturer instructions for automatic corrections or perform manual calculations (SM 4500-O G).

3. PREVENTIVE MAINTENANCE: Refer to FT 1000, section 3.

4. DOCUMENTATION

4.1. Standard and Reagent Documentation: Document information about standards and reagents used for verifications.

4.1.1. Note the date of receipt, the expiration date and the date of first use for all standards and reagents.

4.1.1.1. Document acceptable verification of any standard used after its expiration date.

4.1.2. Record the concentration or other value for the standard in the appropriate measurement units.

4.1.2.1. Note vendor catalog number and description for pre-formulated solutions as well as for neat liquids and powdered standards.

4.1.2.2. Retain vendor assay specifications for standards as part of the calibration record.

4.1.3. Record the grade of standard or reagent used.

4.1.4. When formulated in-house, document all calculations used to formulate calibration standards.

4.1.4.1. Record the date of preparation for all in-house formulations.

4.1.5. Describe or cite the procedure(s) used to prepare any standards in-house (DEP SOP or internal SOP).

4.2. Field Instrument Calibration Documentation: Document acceptable calibration and calibration verification for each instrument unit and field test or analysis, linking this record with affected sample measurements.

4.2.1. Retain vendor certifications of all factory-calibrated instrumentation.

4.2.2. Designate the identity of specific instrumentation in the documentation with a unique description or code for each instrument unit used.

4.2.2.1. Record the manufacturer name, model number and identifying number such as a serial number for each instrument unit.

4.2.3. Record the time and date of all initial calibrations and all calibration verifications.

4.2.4. Record the instrument reading (value in appropriate measurement units) of all calibration verifications.

4.2.5. Record the temperature associated with all calibration verifications.

4.2.6. Record the name of the analyst(s) performing the calibration.

4.2.7. Document the specific standards used to calibrate or verify the instrument or field test with the following information:

- Type of standard or standard name (e.g., saturation)
- Value of standard, including correct units (e.g., mg/L at ^{o}C)
- Link to information recorded according to section 4.1 above

4.2.8. Retain manufacturers' instrument specifications.

4.2.9. Document whether successful initial calibration occurred.

4.2.10. Document whether each calibration verification passed or failed.

4.2.11. Document any corrective actions taken to correct instrument performance according to records requirements of FD 3000.

4.2.11.1. Document the date and time of any corrective action.

4.2.11.2. Note any incidence of discontinuation of use of the instrument due to calibration failure.

4.2.12. Describe or cite the specific calibration or verification procedure performed (DEP SOP or internal SOP).

4.3. Record all field-testing measurement data, to include the following:

- Project name
- Date and time of measurement or test (including time zone, if applicable)
- Source and location of the measurement or test sample (e.g., monitoring well identification number, outfall number, station number or other description)
- Latitude and longitude of sampling source location (if required)
- Analyte or parameter measured
- Measurement or test sample value
- Reporting units
- Initials or name of analyst performing the measurement
- Unique identification of the specific instrument unit(s) used for the test(s)

Appendix FT 1500
Tables, Figures and Forms

Table FT 1500-1: Solubility of Oxygen in Water at Atmospheric Pressure[1,2]

Temperature °C	Oxygen Solubility mg/L	Temperature °C	Oxygen Solubility mg/L
0.0	14.621	26.0	8.113
1.0	14.216	27.0	7.968
2.0	13.829	28.0	7.827
3.0	13.460	29.0	7.691
4.0	13.107	30.0	7.559
5.0	12.770	31.0	7.430
6.0	12.447	32.0	7.305
7.0	12.139	33.0	7.183
8.0	11.843	34.0	7.065
9.0	11.559	35.0	6.950
10.0	11.288	36.0	6.837
11.0	11.027	37.0	6.727
12.0	10.777	38.0	6.620
13.0	10.537	39.0	6.515
14.0	10.306	40.0	6.412
15.0	10.084	41.0	6.312
16.0	9.870	42.0	6.213
17.0	9.665	43.0	6.116
18.0	9.467	44.0	6.021
19.0	9.276	45.0	5.927
20.0	9.092	46.0	5.835
21.0	8.915	47.0	5.744
22.0	8.743	48.0	5.654
23.0	8.578	49.0	5.565
24.0	8.418	50.0	5.477
25.0	8.263		

1. The table provides three decimal places to aid interpolation

2. Under equilibrium conditions, the partial pressure of oxygen in air-saturated water is equal to that of the oxygen in water-saturated

FT 1600. Field Measurement of Turbidity

Use in conjunction with:

- FT 1000 General Field Testing and Measurement
- FS 1000 General Sampling Procedures
- FD 1000 Documentation Procedures

1. INTRODUCTION: Turbidity measures the scattering effect that suspended solids have on the propagation of light through a body of water (surface or ground waters). The higher the effect (i.e., intensity of scattered light), the higher the turbidity value. Suspended and colloidal matter such as clay, silt, finely divided organic and inorganic matter, and plankton and other microscopic organisms cause turbidity in water.

This SOP describes the use of true nephelometric measurement using instruments meeting the specifications outlined in 2.1.

Exceptions to the requirements specified in 2.1 below include:

1.1. *In situ* probes with turbidity sensors used for screening purposes (e.g., groundwater purge stabilization measurements).

1.2. Non standard light sources, detectors or other turbidity measuring devices may be proposed for use in studies that entail comparison measurements (dredge and fill) or unattended deployment for monitoring purposes.

1.3. **Do not report results from "non standard" sensors or configurations for regulatory purposes such as permit compliance unless the Department has approved the use for the specific project.**

1.4. All "non standard" instrument must be calibrated/check according to the principles outlined in this SOP.

2. EQUIPMENT AND SUPPLIES

2.1. Field Instrument: Use a turbidimeter (nephelometer) or a spectrophotometer consisting of a light source and one or more photoelectric detectors with a readout device to indicate the intensity of light. The instrument must meet these specifications:

2.1.1. The light source must have a tungsten-filament lamp operated at a color temperature between 2000 and 3000 K.

2.1.2. The distance traversed by the incident light and scattered light within the sample tube must not exceed 10 cm.

2.1.3. The light detector, positioned at 90^o to the incident light, must have an acceptance angle that does not exceed $\pm 30^o$ from 90^o.

2.1.4. The detector and any filter system must have a spectral peak response between 400 and 600 nanometers.

2.1.5. The instrument sensitivity must permit detection of a turbidity difference of 0.02 NTU at the 0 – 1.0 NTU scale.

2.1.6. Note: using the appropriate equipment and following the procedures in this SOP, the field accuracy of this measurement is close to %R = 100 $\pm$ 10% for turbidities in the range of 1 to 100 NTU.

2.2. Sample Cells (cuvettes): Use sample cells or tubes of clear, colorless glass or plastic.

2.2.1. Keep cells clean, both inside and out, and discard if scratched or etched.

2.2.1.1. Never handle them where the light beam strikes the sample.

2.2.1.2. Clean sample cells by thorough washing with laboratory soap (inside and out) followed by multiple rinses with distilled or de-ionized water, and let air-dry.

2.2.2. Use a very thin layer of silicone oil on the outside surfaces to mask minor imperfections or scratches in the cells.

2.2.2.1. Use silicone oil with the same refractive index of the glass; making sure the cell appear to be nearly dry with little or no visible signs of oil.

2.2.3. Because small differences between cells significantly impact measurement, use either matched pairs or the same cell for standardization and sample measurement.

2.3. Standards:

2.3.1. Primary standards: Use these standards for initial calibration.

2.3.1.1. Formazin standards can be either obtained commercially or prepared according to method SM 2130B, section 3.b. See *Standard Methods for the Examination of Water and Wastewater* (American Public Health Association, American Water Works Association, Water Pollution Control Federation).

2.3.1.2. Some instruments may require the use of styrene divinylbenzene (SDVB) standards for calibration.

2.3.2. Secondary Standards: Use only those certified by the manufacturer for a specific instrument. Secondary standards must only be used for continuing calibration verifications according to the procedures in section 3.4 below. Determine or verify the values of secondary standards according to the procedure in section 3.3 below.

2.3.3. Turbidity-free water: Use filtered, laboratory reagent water demonstrated to be free of measurable turbidity (<0.01 NTU) or purchase commercially prepared turbidity-free water.

3. CALIBRATION AND USE

3.1. General Concerns

3.1.1. Light absorption by dissolved and suspended matter may cause a negative bias on the turbidity measurement. When present in significant concentrations, particles of light-absorbing materials such as activated carbon will cause a negative interference. Likewise, the presence of dissolved, color-causing substances that absorb light may also cause a negative interference. Some commercial instruments may have the capability of either correcting for slight color interference or optically blanking out the color effect.

3.1.2. Handle samples with natural effervescence as described in 3.5.5.1 below.

3.2. Calibration and Initial Calibration Verification

3.2.1. Follow the calibration activities in FT 1000, section 2.2.

3.2.2. Perform an initial calibration using at least two primary standards.

3.2.2.1. If the instrument cannot be calibrated with two standards, calibrate the instrument with one standard and verify with a second standard per 3.2.3 below.

3.2.2.2. For measurement of samples of very low turbidity, select the lowest standard commercially available for bracketing the lower end of the anticipated sample turbidity range or dilute higher turbidity standards with turbidity-free water.

3.2.2.3. Do not use turbidity-free water as a calibration verification standard.

3.2.3. Perform an initial calibration verification by reading at least one primary standard as a sample. The acceptance criterion for the initial calibration verification depends on the range of turbidity of the standard value:

- <u>Standard Value = 0.1-10 NTU:</u> the response must be within 10% of the standard;
- <u>Standard Value = 11-40 NTU:</u> the response must be within 8% of the standard;
- <u>Standard Value = 41-100 NTU:</u> the response must be within 6.5% of the standard; and
- <u>Standard Value > 100 NTU:</u> the response must be within 5% of the standard.

3.3. <u>Determining the Values of Secondary Standards</u>

3.3.1. Use only those standards certified by the manufacturer for a specific instrument.

3.3.2. Use verified secondary standards only for continuing calibration verifications.

3.3.3. Determining the initial value(s) of secondary standard(s):

3.3.3.1. Calibrate or verify the instrument with primary standards. Select primary standards that bracket the range of the secondary standards.

3.3.3.2. Immediately after the an initial calibration with primary standards or verification with a primary standard, read each secondary standard as a sample use the reading from the instrument as the first assigned value.

3.3.4. Verifying Secondary Standards

3.3.4.1. At least once per quarter or at other documented intervals (see 3.3.5 below), determine or verify the values of secondary standards immediately after the instrument has been calibrated or verified with primary standards.

3.3.4.2. Read each secondary standard as a sample. This reading must be within the manufacturer's stated tolerance range and within the acceptance ranges of the assigned standard value as listed in 3.2.3., above. If the criteria in section 3.2.3., above are not met, assign this reading as the value of the standard. If the reading is outside the manufacturer's stated tolerance range, discard the secondary standard.

3.3.5. **More frequent calibration verifications may be required for discharge permit compliance measurements or other regulatory requirements.**

3.4. <u>Continuing Calibration Verification:</u> Perform a continuing calibration verification using at least one primary or secondary standard. The calibration acceptance criteria are the same as those listed in section 3.2.3 above.

3.5. <u>Measuring Turbidity in Samples</u>

3.5.1. Gently agitate the sample and wait until air bubbles disappear.

3.5.2. Double-rinse the sample cell or cuvette with a small amount of the sample. Discard, and pour an aliquot into the sample cell or cuvette.

3.5.3. Gently dry out its external surface with lint-free paper.

3.5.4. Insert the cell in the instrument and read the turbidity directly from the meter display.

3.5.5. Do not use vacuum degassing, ultrasonic bath or other devices to remove bubbles from the sample. If the sample contains visible bubbles or if it effervesces (as in groundwater, with changes in pressure and temperature), make a note of this in the field records and collect a sample for laboratory measurement.

3.5.5.1. If effervescing samples are collected for laboratory analysis collect the sample without leaving headspace in the container and ship it as soon as possible to the laboratory (the holding time for this measurement is only 48 hrs). Ship this sample in wet ice at $4^{o}C$.

3.5.6. Pour out the sample, double-rinse the cuvette with de-ionized water in preparation for the next sample.

4. PREVENTIVE MAINTENANCE: Refer to FT 1000, section 3.

5. DOCUMENTATION

5.1. Standard and Reagent Documentation: Document information about standards and reagents used for calibrations, verifications, and sample measurements.

5.1.1. Note the date of receipt, the expiration date and the date of first use for all standards and reagents.

5.1.1.1. Document acceptable verification of any standard used after its expiration date.

5.1.2. Record the concentration or other value for the standard in the appropriate measurement units.

5.1.2.1. Note vendor catalog number and description for preformulated solutions as well as for neat liquids and powdered standards.

5.1.2.2. Retain vendor assay specifications for standards as part of the calibration record.

5.1.3. Record the grade of standard or reagent used.

5.1.4. When formulated in-house, document all calculations used to formulate calibration standards.

5.1.4.1. Record the date of preparation for all in-house formulations.

5.1.5. Describe or cite the procedure(s) used to prepare any standards in-house (DEP SOP or internal SOP).

5.2. Field Instrument Calibration Documentation: Document acceptable calibration and calibration verification for each instrument unit and field test or analysis, linking this record with affected sample measurements.

5.2.1. Retain vendor certifications of all factory-calibrated instrumentation.

5.2.2. Designate the identity of specific instrumentation in the documentation with a unique description or code for each instrument unit used.

5.2.2.1. Record manufacturer name, model number, and identifying number (such as a serial number) for each instrument unit.

5.2.3. Record the time and date of all initial calibrations and all calibration verifications.

5.2.4. Record the instrument reading (value in appropriate measurement units) of all calibration verifications.

5.2.5. Record the name of the analyst(s) performing the calibration.

5.2.6. Document the specific standards used to calibrate or verify the instrument or field test with the following information:

- Type of standard or standard name (e.g., formazin)
- Value of standard, including correct units (e.g., 20 NTU)
- Link to information recorded according to section 5.1 above

5.2.7. Retain manufacturers' instrument specifications.

5.2.8. Document whether successful initial calibration occurred.

5.2.9. Document whether each calibration verification passed or failed.

5.2.10. Document any corrective actions taken to correct instrument performance according to records requirements of FD 3000.

5.2.10.1. Document date and time of any corrective action.

5.2.10.2. Note any incidence of discontinuation of use of the instrument due to calibration failure.

5.2.11. Describe or cite the specific calibration or verification procedure performed (DEP SOP or internal SOP).

5.3. Record all field-testing measurement data, to include the following:

- Project name
- Date and time of measurement or test (including time zone, if applicable)
- Source and location of the measurement or test sample (e.g., monitoring well identification number, outfall number, station number or other description)
- Latitude and longitude of sampling source location (if required)
- Analyte or parameter measured
- Measurement or test sample value
- Reporting units
- Initials or name of analyst performing the measurement
- Unique identification of the specific instrument unit(s) used for the test(s)

Tetra Tech NUS, Inc.

BORING LOG

PROJECT NAME: ______________ BORING No.: ______________
PROJECT NUMBER: ______________ DATE: ______________
DRILLING COMPANY: ______________ GEOLOGIST: ______________
DRILLING RIG: ______________ DRILLER: ______________

Sample No. and Type or RQD	Depth (Ft.) or Run No.	Blows / 6" or RQD (%)	Sample Recovery / Sample Length	Lithology Change (Depth/Ft.) or Screened Interval	MATERIAL DESCRIPTION: Soil Density/ Consistency or Rock Hardness	MATERIAL DESCRIPTION: Color	MATERIAL DESCRIPTION: Material Classification	U S C S *	Remarks	PID/FID Reading (ppm): Sample	PID/FID Reading (ppm): Sampler BZ	PID/FID Reading (ppm): Borehole**	PID/FID Reading (ppm): Driller BZ**

* When rock coring, enter rock brokeness.

** Include monitor reading in 6 foot intervals @ borehole. Increase reading frequency if elevated reponse read.

Drilling Area Background (ppm): ______

Remarks: ______________________________

Converted to Well: Yes ________ No ________ Well I.D. #: ______________

Tetra Tech NUS, Inc.

PROJECT: ______________________________ JOB #: ____________________
LOCATION: ______________________________ DATE: ____________________
PROJECT MANAGER: ____________________ FOL: __________________________

DAILY ACTIVITIES CHECKLIST

Startup Checklist

Activity	Yes	No	N/A
Pertinent site activities/information entered into site logbook			
All onsite personnel listed in logbook			
Required medical information onsite for all workers (TtNUS and Subcontractors)			
Required MSDS's onsite			
Proper equipment calibrations performed (list equipment)			
1			
2			
3			
4			
Calibration logs filled out			
Tailgate H&S meeting held prior to beginning field activities			
Required work permits filled out/signed			
Required utility clearances obtained			
Required PPE onsite and in use			
Information required to be posted is in place (OSHA poster, hospital route, key phone numbers, etc.)			

Exit Checklist

Activity	Yes	No	N/A
Logbooks completely and comprehensively filled out			
Field forms complete and accounted for/properly filed			
Samples properly packaged/shipped			
COCs faxed to appropriate in-house personnel			
All equipment accounted for, on charge if needed, and properly secured			
All personnel accounted for			
Arrangements made for upcoming work (permits, clearances, equipment, etc.)			
Site properly secured			

Note - not all items listed apply to every job, and some additional requirements may apply on a job-specific basis.

Tetra Tech NUS, Inc.

EQUIPMENT CALIBRATION LOG

PROJECT NAME : ______________________ INSTRUMENT NAME/MODEL: ______________________

SITE NAME: ______________________ MANUFACTURER: ______________________

PROJECT No.: ______________________ SERIAL NUMBER: ______________________

Date of Calibration	Instrument I.D. Number	Person Performing Calibration	Instrument Settings		Instrument Readings		Calibration Standard (Lot No.)	Remarks and Comments
			Pre-calibration	Post-calibration	Pre-calibration	Post-calibration		

Tetra Tech NUS, Inc.

GROUNDWATER LEVEL MEASUREMENT SHEET

Project Name: ______________________ **Project No.:** ______________________
Location: ______________________ **Personnel:** ______________________
Weather Conditions: ______________________ **Measuring Device:** ______________________
Tidally Influenced: Yes ____ No ____ **Remarks:** ______________________

Well or Piezometer Number	Date	Time	Elevation of Reference Point (feet)*	Total Well Depth (feet)*	Water Level Indicator Reading (feet)*	Thickness of Free Product (feet)*	Groundwater Elevation (feet)*	Comments

* All measurements to the nearest 0.01 foot

CHAIN-OF-CUSTODY RECORD

KB LABS Inc.
Mobile Laboratory Services

☐ 25132 SW 1st Avenue
Newberry, FL 32669
TEL (352) 367-0073
FAX (352) 472-5832

☐ 200 Quade Drive
Cary, NC 27513
TEL (919) 678-0030

MOBILE UNIT #

CLIENT NAME	PROJECT NAME & ADDRESS	
SAMPLERS	CONTACT PERSON	**BATCH # (Lab Use Only)**

IDENTIFY PARAMETERS DESIRED AND NO. OF CONTAINERS

PRESERVATION
C Chilled
H HCL
Ot Other (see Remarks)

SAMPLE FIELD ID.\ NUMBER	DATE SAMPLED	TIME SAMPLED	COMP.	GRAB	DATE REC'D	TIME REC'D	STATION LOCATION / No.	SAMPLE MATRIX	NUMBER OF CONTAINERS	VOLATILES				**COMMENT / SAMPLE PRE FIX**

Precleaned Containers Relinquished by: *(Signature)*	Date / Time	Received by: *(Signature)*	Date / Time	**Remarks and Observations**
Relinquished by: *(Signature)*	Date / Time	Received by: *(Signature)*	Date / Time	

Matrix Types S Soil SW Surface Water GW Ground Water SG Soil Gas

Tetra Tech NUS, Inc.

MONITORING WELL DEVELOPMENT RECORD

Site: ____________________________ Depth to Bottom (ft.): _________________ Project Name: ____
Well: ____________________________ Static Water Level Before (ft.): _________ Project Number: ____
Date Installed: ____________________ Static Water Level After (ft.): ___________ Site Geologist: ____
Date Developed: __________________ Screen Length (ft.): __________________ Drilling Co.: ____
Dev. Method: _____________________ Specific Capacity: ___________________
Pump Type: ______________________ Casing ID (in.): _______________________

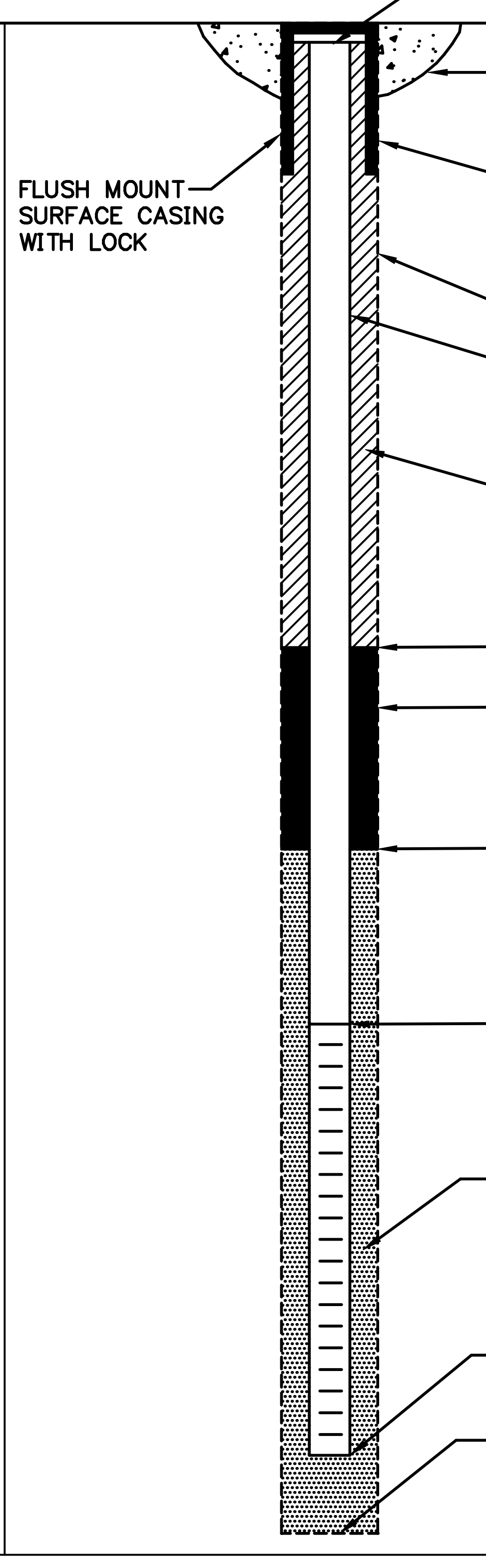
Time	Estimated Sediment Thickness (Ft.)	Cumulative Water Volume (Gal.)	Water Level Readings (Ft. below TOC)	Temperature (Degrees C)	pH	Specific Conductance (Units _____)	Turbidity (NTU)	Remarks (odor, color, etc.)

WELL NO.: ______________

Tetra Tech NUS, Inc.

OVERBURDEN
MONITORING WELL SHEET
FLUSH - MOUNT

PROJECT ______________	LOCATION ______________	DRILLER ______________
PROJECT NO. ______________	BORING ______________	DRILLING METHOD ______________
DATE BEGUN ______________	DATE COMPLETED ______________	DEVELOPMENT METHOD ______________
FIELD GEOLOGIST ______________		
GROUND ELEVATION ______________	DATUM ______________	



ELEVATION TOP OF RISER: ______________

TYPE OF SURFACE SEAL: ______________

TYPE OF PROTECTIVE CASING: ______________

I.D. OF PROTECTIVE CASING: ______________

DIAMETER OF HOLE: ______________

TYPE OF RISER PIPE: ______________

RISER PIPE I.D.: ______________

TYPE OF BACKFILL/SEAL: ______________

ELEVATION/DEPTH TOP OF SEAL: ______ / ______

TYPE OF SEAL: ______________

ELEVATION/DEPTH TOP OF SAND: ______ / ______

ELEVATION/DEPTH TOP OF SCREEN: ______ / ______

TYPE OF SCREEN: ______________

SLOT SIZE x LENGTH: ______________

TYPE OF SAND PACK: ______________

DIAMETER OF HOLE IN BEDROCK: ______________

ELEVATION / DEPTH BOTTOM OF SCREEN: ______ / ______

ELEVATION / DEPTH BOTTOM OF SAND: ______ / ______

ELEVATION/DEPTH BOTTOM OF HOLE: ______ / ______

BACKFILL MATERIAL BELOW SAND: ______________

ACAD: FORM_MWFM.dwg 07/20/99 INL

Tetra Tech NUS, Inc.

PROJECT: ______________________ **LOCATION:** ______________________

JOB & CTO #: ______________________ **MOBILIZATION DATE:** ______________________

PROJECT MANAGER: ______________________ **RETURN DATE:** ______________________

FIELD PROJECT PRE-MOBILIZATION CHECKLIST

TRAVEL

___ Airline reservations
___ Hotel reservations/BOQs
___ Vehicle rental
___ Itinerary
___ Phone/pager number

DRILLING/DPT/SURVEY

Subcontractor

___ POC phone #/address
___ Drill Specification RFP
___ Contact (time & place to meet)
___ Confirm subcontract w/ TtNUS Procurement
___ Health and Safety documentation for all personnel on site
___ Copy of Drillers license
___ Well / boring permits

Utilities (2 weeks lead time)

___ Contact Site POC (Date:____________)
___ Contact Local "Call Before You Dig"
___ Utility Clearance Form

Forms

___ Boring logs / Test Pit logs
___ Well construction / development forms
___ Daily activity forms
___ IDW inventory
___ IDW drum labels
___ Chemical Inventory
___ MSDS's

EQUIPMENT MOBILIZATION

___ Equipment Requisition form completed / equipment ordered
___ 3rd Party rental / misc. equipment ordered
___ Equipment calibration forms
___ Span / calibration gas and regulator

SAMPLING

Forms

___ Sample log sheets
___ Low-flow purge data sheets
___ COC records
___ COC seals
___ Sample labels (from database group)

Laboratory

___ POC address/phone#
___ Order bottles / preservatives
___ Shipping address, also check Sat. address
___ Bottle & preservation req'ts from lab
___ ______________________

MISCELLANEOUS

Schedule

___ Plan field operations w/ Project manager

Documents for Field Program

___ Logbook(s)
___ Field Sampling plan
___ Health & Safety plan
___ Maps
___ H & S Guidance Manual

Authorization

___ Kick-off meeting held
___ Gov't rate letter
___ H&S/OSHA 40-hour certifcate
___ 8-Hour Refresher Training Certificate
___ Medical Clearance Letter
___ Supervisory Training Certificate
___ Health & Safety Clearance Letter
___ Full-size OSHA Poster

HYDROGEOLOGY EQUIPMENT

___ Slug test/pumping test forms
___ Groundwater elevation data sheets
___ Graph paper
___ Data Logger/transducer/data cable
___ Existing well construction & water level data
___ M-Scope, slug

SHIPPING

Forms

___ FedEx Airbills, local dropoff location & hours
___ FedEx Gov. Acct# (1771-8058-0)
___ Lab Shipping Labels
___ Warehouse Shipping Labels
___ Blank Labels

Supplies

___ Tape
___ Packing materials
___ Baggies, Large garbage bags

OTHER

___ Site POC name/phone #
___ Personnel information to POC
___ Mobilization schedule to POC
___ Site access authorizations
___ Field office / trailer arrangements made
___ Electric, phone hookups arranged
___ Steel-toed boots, safety glasses, & hard hat
___ First aid equipment
___ Insect repellent
___ ______________________
___ ______________________

Note - not all items listed apply to every job, and some additional requirements may apply on a job-specific basis.

Tetra Tech NUS, Inc.

QA SAMPLE LOG SHEET

Project Site Name: ____________ Sample ID Number: ____________
Project Number: ____________ Sampled By: ____________
Sample Location: ____________ C.O.C. Number: ____________
QA Sample Type:

[] Trip Blank [] Rinsate Blank
[] Source Water Blank [] Other Blank ____________

SAMPLING DATA:

Date: ____________
Time: ____________
Method: ____________

WATER SOURCE:

[] Laboratory Prepared [] Tap
[] Purchased [] Fire Hydrant
[] Other ____________

PURCHASED WATER INFORMATION (If Applicable as Source or Rinsate Water):

Product Name: ____________
Supplier: ____________
Manufacturer: ____________
Order Number: ____________
Lot Number: ____________
Expiration Date: ____________

RINSATE INFORMATION (If Applicable):

Media Type: ____________
Equipment Used: ____________
Equipment Type:

[] Dedicated
[] Reusable

SAMPLE COLLECTION INFORMATION:

Analysis	Preservative	Container Requirements	Collected
Volatiles	Cool 4°C & HCl		YES / NO
Semivolatiles	Cool 4°C		YES / NO
Pesticide / PCB	Cool 4°C		YES / NO
Metals	Cool 4°C & HNO_3		YES / NO
Cyanide	Cool 4°C & NaOH		YES / NO

OBSERVATIONS / NOTES:

Signature(s):

Tetra Tech NUS, Inc.

SOIL & SEDIMENT SAMPLE LOG SHEET

Page___ of ___

Project Site Name: ______________________

Project No.: ______________________

[] Surface Soil
[] Subsurface Soil
[] Sediment
[] Other: ______________________
[] QA Sample Type: ______________________

Sample ID No.: ______________

Sample Location: ______________

Sampled By: ______________

C.O.C. No.: ______________

Type of Sample:
[] Low Concentration
[] High Concentration

GRAB SAMPLE DATA:

	Depth Interval	Color	Description (Sand, Silt, Clay, Moisture, etc.)
Date:			
Time:			
Method:			
Monitor Reading (ppm):			

COMPOSITE SAMPLE DATA:

	Time	Depth Interval	Color	Description (Sand, Silt, Clay, Moisture, etc.)
Date:				
Method:				
Monitor Readings (Range in ppm):				

SAMPLE COLLECTION INFORMATION:

Analysis	Container Requirements	Collected	Other

OBSERVATIONS / NOTES:

MAP:

Circle if Applicable:

MS/MSD

Duplicate ID No.:

Signature(s):

APPENDIX C

LABORATORY STANDARD OPERATING PROCEDURES

LABORATORY ACCREDITATION BUREAU

Certificate # L2226

Scope of Accreditation For Empirical Laboratories, LLC

621 Mainstream Drive, Suite 270
Nashville, TN 37228
Marcia K. McGinnity
1-877-345-1113

In recognition of a successful assessment to ISO/IEC 17025:2005 and the requirements of the DoD Environmental Laboratory Accreditation Program (DoD ELAP) as detailed in the DoD Quality Systems Manual for Environmental Laboratories (DoD QSM v4.1) based on the National Environmental Laboratory Accreditation Conference Chapter 5 Quality Systems Standard (NELAC Voted Revision June 5, 2003), accreditation is granted to Empirical Laboratories, LLC to perform the following tests:

Accreditation granted through: **November 30, 2012**

Testing - Environmental

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	8260B	1,1,1-Trichloroethane (1,1,1-TCA)
GC/MS	8260B	1,1,2-Trichloro-1,2,2-trifluoroethane (CFC-113; Freon 113)
GC/MS	8260B	1,1,2-Trichloroethane
GC/MS	8260B	1,1,2,2-Tetrachloroethane
GC/MS	8260B	1,1,1,2-Tetrachloroethane
GC/MS	8260B	1,1-Dichloroethane (1,1-DCA)
GC/MS	8260B	1,1-Dichloroethene (1,1-DCE)
GC/MS	8260B	1,2,3-Trichlorobenzene
GC/MS	8260B	1,2,4-Trichlorobenzene
GC/MS	8260B	1,2,3-Trichloropropane
GC/MS	8260B	1,2,4-Trimethylbenzene
GC/MS	8260B	1,3,5-Trimethylbenzene
GC/MS	8260B	1,2-Dibromoethane (EDB)
GC/MS	8260B	1,2-Dibromo-3-chloropropane (DBCP)
GC/MS	8260B	1,2-Dichlorobenzene
GC/MS	8260B	1,2-Dichloroethane (EDC)
GC/MS	8260B	1,2-Dichloropropane
GC/MS	8260B	1,3-Dichlorobenzene

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Technology	Method	Analyte
GC/MS	8260B	1,4-Dichlorobenzene
GC/MS	8260B	1,1-Dichloropropene
GC/MS	8260B	1,3-Dichloropropane
GC/MS	8260B	2,2-Dichloropropane
GC/MS	8260B	2-Butanone (Methyl ethyl ketone; MEK)
GC/MS	8260B	2-Hexanone (Methyl butyl ketone; MBK)
GC/MS	8260B	4-Methyl-2-pentanone (Methyl isobutyl ketone; MIBK)
GC/MS	8260B	Acetone
GC/MS	8260B	Benzene
GC/MS	8260B	Bromochloromethane
GC/MS	8260B	Bromodichloromethane
GC/MS	8260B	Bromobenzene
GC/MS	8260B	Bromoform
GC/MS	8260B	Bromomethane
GC/MS	8260B	n-Butylbenzene
GC/MS	8260B	sec-Butylbenzene
GC/MS	8260B	tert-Butylbenzene
GC/MS	8260B	Carbon Disulfide
GC/MS	8260B	Carbon Tetrachloride
GC/MS	8260B	Chlorobenzene
GC/MS	8260B	Chloroethane
GC/MS	8260B	Chloroform
GC/MS	8260B	Chloromethane
GC/MS	8260B	2-Chlorotoluene
GC/MS	8260B	4-Chlorotoluene
GC/MS	8260B	cis-1,2-Dichloroethene (cis-1,2-DCE)
GC/MS	8260B	cis-1,3-Dichloropropene
GC/MS	8260B	Cyclohexane
GC/MS	8260B	Dibromochloromethane
GC/MS	8260B	Dibromomethane
GC/MS	8260B	Dichlorodifluoromethane (CFC-12)
GC/MS	8260B	Ethylbenzene
GC/MS	8260B	Hexachlorobutadiene
GC/MS	8260B	Isopropylbenzene (Cumene)
GC/MS	8260B	p-Isopropyltoluene
GC/MS	8260B	Methyl Acetate
GC/MS	8260B	Methyl Tertiary Butyl Ether (MTBE)
GC/MS	8260B	Methylcyclohexane
GC/MS	8260B	Methylene Chloride, or Dichloromethane

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Non-Potable Water

Technology	Method	Analyte
GC/MS	8260B	Naphthalene
GC/MS	8260B	n-Propylbenzene
GC/MS	8260B	Styrene
GC/MS	8260B	Tetrachloroethene (PCE; PERC)
GC/MS	8260B	Toluene
GC/MS	8260B	trans-1,2-Dichloroethene (trans-1,2-DCE)
GC/MS	8260B	trans-1,3-Dichloropropene
GC/MS	8260B	Trichloroethene (TCE)
GC/MS	8260B	Trichlorofluoromethane (CFC-11)
GC/MS	8260B	Vinyl Chloride (VC)
GC/MS	8260B	Xylenes (Total)
GC/MS	8260B	Acrolein
GC/MS	8260B	Acrylonitrile
GC/MS	8260B	Di-isopropyl ether
GC/MS	8260B	ETBE
GC/MS	8260B	Ethyl methacrylate
GC/MS	8260B	Iodomethane
GC/MS	8260B	Methyl methacrylate
GC/MS	8260B	t-Butyl alcohol
GC/MS	8260B	tert-Amyl methyl ether
GC/MS	8260B	Vinyl acetate
GC/MS	8270C/D	Bis(2-chloroisopropyl)ether, or 2,2'-oxybis (1-Chloropropane)
GC/MS	8270C/D	1,2-Dichlorobenzene
GC/MS	8270C/D	1,3-Dichlorobenzene
GC/MS	8270C/D	1,4-Dichlorobenzene
GC/MS	8270C/D	2,4,5-Trichlorophenol
GC/MS	8270C/D	2,4,6-Trichlorophenol (TCP)
GC/MS	8270C/D	2,4-Dichlorophenol (DCP)
GC/MS	8270C/D	2,4-Dimethylphenol
GC/MS	8270C/D	2,4-Dinitrophenol
GC/MS	8270C/D	2,4-Dinitrotoluene (DNT)
GC/MS	8270C/D	2,6-Dichlorophenol
GC/MS	8270C/D	2,6-Dinitrotoluene
GC/MS	8270C/D	1,2-Diphenylhydrazine
GC/MS	8270C/D	2-Chloronaphthalene
GC/MS	8270C/D	2-Chlorophenol
GC/MS	8270C/D	2-Methylnaphthalene
GC/MS	8270C/D	2-Methylphenol (o-Cresol)
GC/MS	8270C/D	2-Nitroaniline

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Non-Potable Water

Technology	Method	Analyte
GC/MS	8270C/D	2-Nitrophenol (ONP)
GC/MS	8270C/D	3,3'-Dichlorobenzidine (DCB)
GC/MS	8270C/D	3-Methylphenol
GC/MS	8270C/D	3-Nitroaniline
GC/MS	8270C/D	4,6-Dinitro-2-methylphenol (DNOC)
GC/MS	8270C/D	4-Bromophenyl phenyl ether
GC/MS	8270C/D	4-Chloro-3-methylphenol
GC/MS	8270C/D	4-Chloroaniline
GC/MS	8270C/D	4-Chlorophenyl phenyl ether
GC/MS	8270C/D	4-Methylphenol (p-Cresol)
GC/MS	8270C/D	4-Nitroaniline (PNA)
GC/MS	8270C/D	4-Nitrophenol (PNP)
GC/MS	8270C/D	Acenaphthene
GC/MS	8270C/D	Acenaphthylene
GC/MS	8270C/D	Acetaphenone
GC/MS	8270C/D	Anthracene
GC/MS	8270C/D	Benzo(a)anthracene
GC/MS	8270C/D	Benzo(a)pyrene
GC/MS	8270C/D	Benzo(b)fluoranthene
GC/MS	8270C/D	Benzo(g,h,i)perylene
GC/MS	8270C/D	Benzo(k)fluoranthene
GC/MS	8270C/D	Benzyl alcohol
GC/MS	8270C/D	Benzoic Acid
GC/MS	8270C/D	bis(2-Chloroethoxy)methane
GC/MS	8270C/D	bis(2-Chloroethyl)ether (BCEE)
GC/MS	8270C/D	bis(2-Ethylhexyl)phthalate (BEHP)
GC/MS	8270C/D	Butyl benzyl phthalate (BBP)
GC/MS	8270C/D	Carbazole
GC/MS	8270C/D	Chrysene
GC/MS	8270C/D	Di-n-butyl phthalate (DBP)
GC/MS	8270C/D	Di-n-octyl phthalate (DNOP)
GC/MS	8270C/D	Dibenz(a,h)anthracene
GC/MS	8270C/D	Dibenzofuran (DBF)
GC/MS	8270C/D	Diethyl phthalate (DEP)
GC/MS	8270C/D	Dimethyl phthalate (DMP)
GC/MS	8270C/D	Fluoranthene
GC/MS	8270C/D	Fluorene
GC/MS	8270C/D	Hexachlorobenzene (HCB)
GC/MS	8270C/D	Hexachlorobutadiene (HCBD)

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Non-Potable Water

Technology	Method	Analyte
GC/MS	8270C/D	Hexachlorocyclopentadiene (HCCPD)
GC/MS	8270C/D	Hexachloroethane (HCE)
GC/MS	8270C/D	Indeno(1,2,3-cd)pyrene
GC/MS	8270C/D	Isophorone
GC/MS	8270C/D	N-Nitrosodimethylamine
GC/MS	8270C/D	N-Nitroso-di-n-propylamine (NDPA)
GC/MS	8270C/D	N-nitrosodiphenylamine (NDPHA)
GC/MS	8270C/D	Naphthalene
GC/MS	8270C/D	Nitrobenzene
GC/MS	8270C/D	Pentachlorophenol
GC/MS	8270C/D	Phenanthrene
GC/MS	8270C/D	Phenol
GC/MS	8270C/D	Pyrene
GC/MS	8270C/D	Pyridine
GC/MS	8270C/D	1,2,4-Trichlorobenzene
GC/MS	8270C/D	1,1'-Biphenyl
GC/MS	8270C/D	1,2,4,5-Tetrachlorobenzene
GC/MS	8270C/D	1,4-Dioxane
GC/MS	8270C/D	1-Methylnaphthalene
GC/MS	8270C/D	2,3,4,6-Tetrachlorophenol
GC/MS	8270C/D	Aniline
GC/MS	8270C/D	Atrazine
GC/MS	8270C/D	Benzaldehyde
GC/MS	8270C/D	Benzidine
GC/MS	8270C/D	Caprolactam
GC/ECD	8081A/B	4,4'-DDD
GC/ECD	8081A/B	4,4'-DDE
GC/ECD	8081A/B	4,4'-DDT
GC/ECD	8081A/B	Aldrin
GC/ECD	8081A/B	alpha-BHC (alpha-HCH)
GC/ECD	8081A/B	alpha-Chlordane
GC/ECD	8081A/B	beta-BHC (beta-HCH)
GC/ECD	8081A/B	delta-BHC (delta-HCH)
GC/ECD	8081A/B	Dieldrin
GC/ECD	8081A/B	Endosulfan I
GC/ECD	8081A/B	Endosulfan II
GC/ECD	8081A/B	Endosulfan sulfate
GC/ECD	8081A/B	Endrin

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Non-Potable Water

Technology	Method	Analyte
GC/ECD	8081A/B	Endrin aldehyde
GC/ECD	8081A/B	Endrin ketone
GC/ECD	8081A/B	gamma-BHC (Lindane; gamma-HCH)
GC/ECD	8081A/B	gamma-Chlordane
GC/ECD	8081A/B	Heptachlor
GC/ECD	8081A/B	Heptachlor epoxide
GC/ECD	8081A/B	Methoxychlor
GC/ECD	8081A/B	Chlordane
GC/ECD	8081A/B	Toxaphene
GC/ECD	8082 /A	Aroclor-1016
GC/ECD	8082 /A	Aroclor-1221
GC/ECD	8082 /A	Aroclor-1232
GC/ECD	8082 /A	Aroclor-1242
GC/ECD	8082 /A	Aroclor-1248
GC/ECD	8082 /A	Aroclor-1254
GC/ECD	8082 /A	Aroclor-1260
GC/ECD	8151A	2,4,5-T
GC/ECD	8151A	2,4,5-TP (Silvex)
GC/ECD	8151A	2,4-D
GC/ECD	8151A	2,4-DB
GC/ECD	8151A	Dalapon
GC/ECD	8151A	Dicamba
GC/ECD	8151A	Dichlorprop
GC/ECD	8151A	Dinoseb
GC/ECD	8151A	MCPA
GC/ECD	8151A	MCPP (Mecoprop)
HPLC/UV	8330A	1,3,5-Trinitrobenzene
HPLC/UV	8330A	1,3-Dinitrobenzene
HPLC/UV	8330A	2,4,6-Trinitrophenylmethylnitramine (Tetryl)
HPLC/UV	8330A	2,4,6-Trinitrotoluene (TNT)
HPLC/UV	8330A	2,4-Dinitrotoluene (DNT)
HPLC/UV	8330A	2,6-Dinitrotoluene
HPLC/UV	8330A	2-Amino-4,6-dinitrotoluene
HPLC/UV	8330A	2-Nitrotoluene (ONT)
HPLC/UV	8330A	3-Nitrotoluene
HPLC/UV	8330A	4-Amino-2,6-dinitrotoluene
HPLC/UV	8330A	4-Nitrotoluene (PNT)
HPLC/UV	8330A	Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)
HPLC/UV	8330A	Nitroglycerin

Non-Potable Water

Technology	Method	Analyte
HPLC/UV	8330A	Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)
HPLC/UV	8330A	3,5-Dinitroaniline
HPLC/UV	8330A	PETN
GC/FID	8015B	TPH DRO
GC/FID	8015B	TPH GRO
GC/FID	RSK-175	Methane
GC/FID	RSK-175	Ethane
GC/FID	RSK-175	Ethene
GC/ECD	8011	1,2-Dibromoethane (EDB)
GC/ECD	8011	1,2-Dibromo-3-chloropropane (DBCP)
HPLC/MS	6850	Perchlorate
ICP	6010B/C	Aluminum
ICP	6010B/C	Antimony
ICP	6010B/C	Arsenic
ICP	6010B/C	Barium
ICP	6010B/C	Beryllium
ICP	6010B/C	Cadmium
ICP	6010B/C	Calcium
ICP	6010B/C	Chromium, total
ICP	6010B/C	Cobalt
ICP	6010B/C	Copper
ICP	6010B/C	Iron
ICP	6010B/C	Lead
ICP	6010B/C	Magnesium
ICP	6010B/C	Manganese
CVAA	7470A	Mercury
ICP	6010B/C	Nickel
ICP	6010B/C	Potassium
ICP	6010B/C	Selenium
ICP	6010B/C	Silver
ICP	6010B/C	Sodium
ICP	6010B/C	Thallium
ICP	6010B/C	Vanadium
ICP	6010B/C	Zinc
ICP	6010B/C	Molybdenum
ICP	6010B/C	Tin
ICP	6010B/C	Titanium
IC	300.0	Chloride
IC	300.0	Fluoride

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Non-Potable Water		
Technology	**Method**	**Analyte**
IC	300.0	Nitrate
IC	300.0	Nitrite
IC	300.0	Sulfate
IC	9056A	Chloride
IC	9056A	Fluoride
IC	9056A	Nitrate
IC	9056A	Nitrite
IC	9056A	Sulfate
Titration	SM 2320B 20th ed.	Alkalinity
ISE	SM 4500 B, D, 20th ed.	Ammonia
UV/Vis	7196A	Hexavalent Chromium
Colorimetric	353.2	Nitrate/Nitrite
Titration	Chap.7, Sect. 7.3.4 Mod.	Reactive Sulfide
Titration	SM 4500 S-2CF, 20th edition	Sulfide
UV/Vis	SM 4500 P B5, E, 20th edition	Total Phosphorus
UV/Vis	SM 4500 PE, 20th edition	Ortho-Phosphorus
TOC	9060A/SM5310C, 20th edition	Total Organic Carbon
Gravimetric	SM 2540C, 20th edition	TDS
Colorimetric	9012A/B	Cyanide
Physical	1010A	Ignitability
Physical	9095B	Paint Filter
Probe	9040B/C	pH
Preparation	**Method**	**Type**
Preparation	1311	TCLP
Preparation	3005A	Metals digestion
Preparation	3010A	Metals digestion
Preparation	3510C	Organics Liquid Extraction
Preparation	5030A/B	Purge and Trap Water

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS	8260B	1,1,1-Trichloroethane (1,1,1-TCA)
GC/MS	8260B	1,1,2-Trichloro-1,2,2-trifluoroethane (CFC-113; Freon 113)
GC/MS	8260B	1,1,2-Trichloroethane
GC/MS	8260B	1,1,2,2-Tetrachloroethane
GC/MS	8260B	1,1,1,2-Tetrachloroethane
GC/MS	8260B	1,1-Dichloroethane (1,1-DCA)
GC/MS	8260B	1,1-Dichloroethene (1,1-DCE)
GC/MS	8260B	1,2,3-Trichlorobenzene
GC/MS	8260B	1,2,4-Trichlorobenzene
GC/MS	8260B	1,2,3-Trichloropropane
GC/MS	8260B	1,2,4-Trimethylbenzene
GC/MS	8260B	1,3,5-Trimethylbenzene
GC/MS	8260B	1,2-Dibromoethane (EDB)
GC/MS	8260B	1,2-Dibromo-3-chloropropane (DBCP)
GC/MS	8260B	1,2-Dichlorobenzene
GC/MS	8260B	1,2-Dichloroethane (EDC)
GC/MS	8260B	1,2-Dichloropropane
GC/MS	8260B	1,3-Dichlorobenzene
GC/MS	8260B	1,4-Dichlorobenzene
GC/MS	8260B	1,1-Dichloropropene
GC/MS	8260B	1,3-Dichloropropane
GC/MS	8260B	2,2-Dichloropropane
GC/MS	8260B	2-Butanone (Methyl ethyl ketone; MEK)
GC/MS	8260B	2-Hexanone (Methyl butyl ketone; MBK)
GC/MS	8260B	4-Methyl-2-pentanone (Methyl isobutyl ketone; MIBK)
GC/MS	8260B	Acetone
GC/MS	8260B	Benzene
GC/MS	8260B	Bromochloromethane
GC/MS	8260B	Bromodichloromethane
GC/MS	8260B	Bromobenzene
GC/MS	8260B	Bromoform
GC/MS	8260B	Bromomethane
GC/MS	8260B	n-Butylbenzene
GC/MS	8260B	sec-Butylbenzene
GC/MS	8260B	tert-Butylbenzene
GC/MS	8260B	Carbon Disulfide
GC/MS	8260B	Carbon Tetrachloride
GC/MS	8260B	Chlorobenzene
GC/MS	8260B	Chloroethane

Solid and Chemical Materials

Technology	Method	Analyte
GC/MS	8260B	Chloroform
GC/MS	8260B	Chloromethane
GC/MS	8260B	2-Chlorotoluene
GC/MS	8260B	4-Chlorotoluene
GC/MS	8260B	cis-1,2-Dichloroethene (cis-1,2-DCE)
GC/MS	8260B	cis-1,3-Dichloropropene
GC/MS	8260B	Cyclohexane
GC/MS	8260B	Dibromochloromethane
GC/MS	8260B	Dibromomethane
GC/MS	8260B	Dichlorodifluoromethane (CFC-12)
GC/MS	8260B	Ethylbenzene
GC/MS	8260B	Hexachlorobutadiene
GC/MS	8260B	Isopropylbenzene (Cumene)
GC/MS	8260B	p-Isopropyltoluene
GC/MS	8260B	Methyl Acetate
GC/MS	8260B	Methyl Tertiary Butyl Ether (MTBE)
GC/MS	8260B	Methylcyclohexane
GC/MS	8260B	Methylene Chloride, or Dichloromethane
GC/MS	8260B	Naphthalene
GC/MS	8260B	n-Propylbenzene
GC/MS	8260B	Styrene
GC/MS	8260B	Tetrachloroethene (PCE; PERC)
GC/MS	8260B	Toluene
GC/MS	8260B	trans-1,2-Dichloroethene (trans-1,2-DCE)
GC/MS	8260B	trans-1,3-Dichloropropene
GC/MS	8260B	Trichloroethene (TCE)
GC/MS	8260B	Trichlorofluoromethane (CFC-11)
GC/MS	8260B	Vinyl Chloride (VC)
GC/MS	8260B	Xylenes (Total)
GC/MS	8260B	Acrolein
GC/MS	8260B	Acrylonitrile
GC/MS	8260B	Ethyl methacrylate
GC/MS	8260B	Iodomethane
GC/MS	8260B	Methyl methacrylate
GC/MS	8260B	Vinyl acetate
GC/MS	8270C/D	Bis(2-chloroisopropyl)ether, or 2,2'-oxybis (1-Chloropropane)
GC/MS	8270C/D	1,2-Dichlorobenzene
GC/MS	8270C/D	1,3-Dichlorobenzene
GC/MS	8270C/D	1,4-Dichlorobenzene

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Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS	8270C/D	2,4,5-Trichlorophenol
GC/MS	8270C/D	2,4,6-Trichlorophenol (TCP)
GC/MS	8270C/D	2,4-Dichlorophenol (DCP)
GC/MS	8270C/D	2,4-Dimethylphenol
GC/MS	8270C/D	2,4-Dinitrophenol
GC/MS	8270C/D	2,4-Dinitrotoluene (DNT)
GC/MS	8270C/D	2,6-Dichlorophenol
GC/MS	8270C/D	2,6-Dinitrotoluene
GC/MS	8270C/D	1,2-Diphenylhydrazine
GC/MS	8270C/D	2-Chloronaphthalene
GC/MS	8270C/D	2-Chlorophenol
GC/MS	8270C/D	2-Methylnaphthalene
GC/MS	8270C/D	2-Methylphenol (o-Cresol)
GC/MS	8270C/D	2-Nitroaniline
GC/MS	8270C/D	2-Nitrophenol (ONP)
GC/MS	8270C/D	3,3'-Dichlorobenzidine (DCB)
GC/MS	8270C/D	3-Methylphenol
GC/MS	8270C/D	3-Nitroaniline
GC/MS	8270C/D	4,6-Dinitro-2-methylphenol (DNOC)
GC/MS	8270C/D	4-Bromophenyl phenyl ether
GC/MS	8270C/D	4-Chloro-3-methylphenol
GC/MS	8270C/D	4-Chloroaniline
GC/MS	8270C/D	4-Chlorophenyl phenyl ether
GC/MS	8270C/D	4-Methylphenol (p-Cresol)
GC/MS	8270C/D	4-Nitroaniline (PNA)
GC/MS	8270C/D	4-Nitrophenol (PNP)
GC/MS	8270C/D	Acenaphthene
GC/MS	8270C/D	Acenaphthylene
GC/MS	8270C/D	Acetaphenone
GC/MS	8270C/D	Anthracene
GC/MS	8270C/D	Benzo(a)anthracene
GC/MS	8270C/D	Benzo(a)pyrene
GC/MS	8270C/D	Benzo(b)fluoranthene
GC/MS	8270C/D	Benzo(g,h,i)perylene
GC/MS	8270C/D	Benzo(k)fluoranthene
GC/MS	8270C/D	Benzyl alcohol
GC/MS	8270C/D	Benzoic Acid
GC/MS	8270C/D	bis(2-Chloroethoxy)methane
GC/MS	8270C/D	bis(2-Chloroethyl)ether (BCEE)

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS	8270C/D	bis(2-Ethylhexyl)phthalate (BEHP)
GC/MS	8270C/D	Butyl benzyl phthalate (BBP)
GC/MS	8270C/D	Carbazole
GC/MS	8270C/D	Chrysene
GC/MS	8270C/D	Di-n-butyl phthalate (DBP)
GC/MS	8270C/D	Di-n-octyl phthalate (DNOP)
GC/MS	8270C/D	Dibenz(a,h)anthracene
GC/MS	8270C/D	Dibenzofuran (DBF)
GC/MS	8270C/D	Diethyl phthalate (DEP)
GC/MS	8270C/D	Dimethyl phthalate (DMP)
GC/MS	8270C/D	Fluoranthene
GC/MS	8270C/D	Fluorene
GC/MS	8270C/D	Hexachlorobenzene (HCB)
GC/MS	8270C/D	Hexachlorobutadiene (HCBD)
GC/MS	8270C/D	Hexachlorocyclopentadiene (HCCPD)
GC/MS	8270C/D	Hexachloroethane (HCE)
GC/MS	8270C/D	Indeno(1,2,3-cd)pyrene
GC/MS	8270C/D	Isophorone
GC/MS	8270C/D	N-Nitrosodimethylamine
GC/MS	8270C/D	N-Nitroso-di-n-propylamine (NDPA)
GC/MS	8270C/D	N-nitrosodiphenylamine (NDPHA)
GC/MS	8270C/D	Naphthalene
GC/MS	8270C/D	Nitrobenzene
GC/MS	8270C/D	Pentachlorophenol
GC/MS	8270C/D	Phenanthrene
GC/MS	8270C/D	Phenol
GC/MS	8270C/D	Pyrene
GC/MS	8270C/D	Pyridine
GC/MS	8270C/D	1,2,4-Trichlorobenzene
GC/MS	8270C/D	1,1'-Biphenyl
GC/MS	8270C/D	1,2,4,5-Tetrachlorobenzene
GC/MS	8270C/D	1,4-Dioxane
GC/MS	8270C/D	1-Methylnaphthalene
GC/MS	8270C/D	2,3,4,6-Tetrachlorophenol
GC/MS	8270C/D	Aniline
GC/MS	8270C/D	Atrazine
GC/MS	8270C/D	Benzaldehyde
GC/MS	8270C/D	Benzidine
GC/MS	8270C/D	Caprolactam

Solid and Chemical Materials

Technology	Method	Analyte
GC/ECD	8081A/B	4,4'-DDD
GC/ECD	8081A/B	4,4'-DDE
GC/ECD	8081A/B	4,4'-DDT
GC/ECD	8081A/B	Aldrin
GC/ECD	8081A/B	alpha-BHC (alpha-HCH)
GC/ECD	8081A/B	alpha-Chlordane
GC/ECD	8081A/B	beta-BHC (beta-HCH)
GC/ECD	8081A/B	delta-BHC (delta-HCH)
GC/ECD	8081A/B	Dieldrin
GC/ECD	8081A/B	Endosulfan I
GC/ECD	8081A/B	Endosulfan II
GC/ECD	8081A/B	Endosulfan sulfate
GC/ECD	8081A/B	Endrin
GC/ECD	8081A/B	Endrin aldehyde
GC/ECD	8081A/B	Endrin ketone
GC/ECD	8081A/B	gamma-BHC (Lindane; gamma-HCH)
GC/ECD	8081A/B	gamma-Chlordane
GC/ECD	8081A/B	Heptachlor
GC/ECD	8081A/B	Heptachlor epoxide
GC/ECD	8081A/B	Methoxychlor
GC/ECD	8081A/B	Chlordane
GC/ECD	8081A/B	Toxaphene
GC/ECD	8082 /A	Aroclor-1016
GC/ECD	8082 /A	Aroclor-1221
GC/ECD	8082 /A	Aroclor-1232
GC/ECD	8082 /A	Aroclor-1242
GC/ECD	8082 /A	Aroclor-1248
GC/ECD	8082 /A	Aroclor-1254
GC/ECD	8082 /A	Aroclor-1260
GC/ECD	8151A	2,4,5-T
GC/ECD	8151A	2,4,5-TP (Silvex)
GC/ECD	8151A	2,4-D
GC/ECD	8151A	2,4-DB
GC/ECD	8151A	Dalapon
GC/ECD	8151A	Dicamba
GC/ECD	8151A	Dichlorprop
GC/ECD	8151A	Dinoseb
GC/ECD	8151A	MCPA
GC/ECD	8151A	MCPP (Mecoprop)

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Certificate # L2226

Solid and Chemical Materials

Technology	Method	Analyte
HPLC/UV	8330A	1,3,5-Trinitrobenzene
HPLC/UV	8330A	1,3-Dinitrobenzene
HPLC/UV	8330A	2,4,6-Trinitrophenylmethylnitramine (Tetryl)
HPLC/UV	8330A	2,4,6-Trinitrotoluene (TNT)
HPLC/UV	8330A	2,4-Dinitrotoluene (DNT)
HPLC/UV	8330A	2,6-Dinitrotoluene
HPLC/UV	8330A	2-Amino-4,6-dinitrotoluene
HPLC/UV	8330A	2-Nitrotoluene (ONT)
HPLC/UV	8330A	3-Nitrotoluene
HPLC/UV	8330A	4-Amino-2,6-dinitrotoluene
HPLC/UV	8330A	4-Nitrotoluene (PNT)
HPLC/UV	8330A	Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)
HPLC/UV	8330A	Nitroglycerin
HPLC/UV	8330A	Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)
HPLC/UV	8330A	PETN
GC/FID	8015B	TPH DRO
GC/FID	8015B	TPH GRO
HPLC/MS	6850	Perchlorate
ICP	6010B/C	Aluminum
ICP	6010B/C	Antimony
ICP	6010B/C	Arsenic
ICP	6010B/C	Barium
ICP	6010B/C	Beryllium
ICP	6010B/C	Cadmium
ICP	6010B/C	Calcium
ICP	6010B/C	Chromium, total
ICP	6010B/C	Cobalt
ICP	6010B/C	Copper
ICP	6010B/C	Iron
ICP	6010B/C	Lead
ICP	6010B/C	Magnesium
ICP	6010B/C	Manganese
CVAA	7471A/B	Mercury
ICP	6010B/C	Nickel
ICP	6010B/C	Potassium
ICP	6010B/C	Selenium
ICP	6010B/C	Silver
ICP	6010B/C	Sodium
ICP	6010B/C	Thallium

LABORATORY ACCREDITATION BUREAU

Certificate # L2226

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
ICP	6010B/C	Vanadium
ICP	6010B/C	Zinc
ICP	6010B/C	Molybdenum
ICP	6010B/C	Tin
ICP	6010B/C	Titanium
UV/Vis	7196A	Hexavalent Chromium
TOC	Lloyd Kahn	Total Organic Carbon
Colorimetric	9012A/B	Cyanide
Titration	Chap.7, Sect. 7.3.4 Mod.	Reactive Sulfide
Titration	9034	Sulfide
Probe	9045D	pH
Preparation	**Method**	**Type**
Preparation	1311	TCLP
Preparation	1312	SPLP
Preparation	NJ Modified 3060A	Hexavalent Chromium
Preparation	3050B	Metals Digestion
Preparation	3546	Organics Microwave Extraction
Preparation	3541	Organics Soxhlet Extraction
Preparation	3550B	Organics Sonication
Preparation	SM 2540B 20th edition	Percent Solids (Percent Moisture)
Preparation	5035 /A	Purge and Trap Solid

Notes:

1) This laboratory offers commercial testing service.

Approved By: ______________________ Date: November 30, 2009

R. Douglas Leonard
Chief Technical Officer

Issued: 11/30/09

	Analyte	QC Limits
Water	1-Methylnaphthalene	35-131
Water	2-Methylnaphthalene	36-121
Water	Acenaphthene	41-132
Water	Acenaphthylene	43-140
Water	Anthracene	50-139
Water	Benzo (a) anthracene	58-141
Water	Benzo (a) pyrene	31-142
Water	Benzo (b) fluoranthene	42-156
Water	Benzo (g,h,i) perylene	12-171
Water	Benzo (k) fluoranthene	49-165
Water	Chrysene	51-155
Water	Dibenz (a,h) anthracene	28-153
Water	Fluoranthene	47-158
Water	Fluorene	40-140
Water	Indeno (1,2,3-cd) pyrene	20-167
Water	Naphthalene	39-125
Water	Phenanthrene	46-144
Water	Pyrene	39-158
Water	2-Fluorobiphenyl	34-167
Water	Terphenyl-d14	34-167
Solid	1-Methylnaphthalene	30-111
Solid	2-Methylnaphthalene	30-111
Solid	Acenaphthene	28-110
Solid	Acenaphthylene	23-126
Solid	Anthracene	28-136
Solid	Benzo (a) anthracene	31-146
Solid	Benzo (a) pyrene	28-128
Solid	Benzo (b) fluoranthene	30-139
Solid	Benzo (g,h,i) perylene	21-149
Solid	Benzo (k) fluoranthene	42-129
Solid	Chrysene	39-134
Solid	Dibenz (a,h) anthracene	30-138
Solid	Fluoranthene	30-142
Solid	Fluorene	27-116
Solid	Indeno (1,2,3-cd) pyrene	17-164
Solid	Naphthalene	29-106
Solid	Phenanthrene	32-127
Solid	Pyrene	28-130
Solid	2-Fluorobiphenyl	14-129
Solid	Terphenyl-d14	14-129

EMPIRICAL LABORATORIES, LLC
STANDARD OPERATING PROCEDURE

ORGANICS: SOP100 REVISION #: 20 EFFECTIVE DATE: 042710

METALS DIGESTION/PREPARATION

References:

Methods 3005A/USEPA CLPILM0 4.1 Aqueous, 3010A, 3030C, 3050B
USEPA CLPILM0 4.1 (Soil/Sediment), 200.7, Standard Methods 3030C
See Addendum for USEPA CLPILM 05.2 (Aqueous & Soil/Sediment)

APPROVALS:

Lab Director: D. R. D. Date: 4/28/10

Data Quality Manager: Marcia Date: 4/28/10

Section Supervisor: Betty DeVille Date: 4/28/10

Changes Summary

Revision 20, 4/27/10

- The SOP is an update from Revision 19 dated 04/20/09.
- References to oil sample preparation have been removed.
- Extraction volumes for TCLP have been updated.

METALS DIGESTION/PREPARATION

References:
Methods 3005A/USEPA CLPILM0 4.1 Aqueous, 3010A, 3030C, 3050B
USEPA CLPILM0 4.1 (Soil/Sediment), 200.7, Standard Methods 3030C
See Addendum for USEPA CLPILM 05.2 (Aqueous & Soil/Sediment)

I. SCOPE AND APPLICATION

A. AQUEOUS
   1. Method 3005A and USEPA CLP ILM0 4.1, "Acid Digestion of Waters for Total Recoverable or Dissolved Metals for Analysis by ICP Spectroscopy".
      a. This method is used to prepare surface water, ground water, drinking water and wastewater samples for analysis by inductively coupled argon plasma spectroscopy (ICP).
   2. Method 200.7, "Determination of Metals and Trace Metals in Water and Wastes by Inductively Coupled Plasma-Atomic Emission Spectrometry"
      a. This method is used to prepare surface water, ground water, drinking water and wastewater samples for analysis by inductively coupled argon plasma spectroscopy (ICP).
   3. Method 3010A, "Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by ICP Spectroscopy".
      a. This method is used to prepare aqueous samples, EP and mobility-procedure extracts, and wastes that contain suspended solids for analysis by ICP. The procedure is used to determine total metals.
   4. Method 3030C (Standard methods), "Preliminary Treatment for Acid-Extractable Metals".
      a. This method is used to prepare ground water samples from North Carolina for analysis by ICP.

B. SOLIDS
   1. Method 3050B, "Acid Digestion of Sediments, Sludges and Soils".
      a. This method is used to prepare sediments, sludges and soil samples for analysis by ICP. Since certain matrices may result in poor recovery, the method of standard additions may be used when analyzed.
      b. It should be noted that some metals could be biased high with the soil digestion when dilution is necessary. Take necessary measures to ensure that dilutions are made as accurately as possible.
   2. USEPA CLP ILM0 4.1, "Acid Digestion of Soil/Sediment"
      a. This method is used to prepare sediments and soil samples for analysis by ICP. Since certain matrices may result in poor recovery, the method of standard additions may be used when analyzed.

D. **NOTES:**
   1. "Total Metals" includes all metals, inorganically and organically bound and both dissolved and particulate.
   2. "Dissolved metals" includes all metals present in a sample after filtration through a 0.45 micron filter followed by digestion.

II. SUMMARY OF METHODS

A. A representative sample of water or soil is put into an acid medium and exposed to heat for a certain amount of time. This allows for reduction of interferences by organic matter and converts metals bound to particulates to form the free metal that can be determined by ICP-Atomic Emission Spectrometry.

NOTE: When a reporting limit is required for a project lower than is customary, a four times concentration or alternate soil digestion ratio must be used in order to reach that lower level. Care must be taken to matrix match this concentrated aliquot. A blank and laboratory control sample (at a reduced concentration) are required with this concentration. A matrix spike (not at reduced concentration) and duplicate or matrix spike and matrix spike duplicate is needed per 20 samples or per batch.

III. SAMPLE HANDLING AND PRESERVATION

A. AQUEOUS
   1. Samples are taken in high density polyethylene, one liter bottles. Samples should be preserved with concentrated HNO_3 to a pH <2 immediately once sampled. If dissolved metals are to be analyzed the sample should be filtered before the HNO_3 is added. The samples should be maintained at 4°C until analysis. The holding time for metals samples is 180 days or approximately 6 months.

B. SOLIDS
   1. Samples are taken in high density polyethylene(CLP only) or glass bottles. The samples should be maintained at 4°C until analysis. The holding time for metals samples is 180 days or approximately 6 months.

IV. INTERFERENCES

A. AQUEOUS
   1. Solvents, reagents, glassware, and other sample processing hardware may yield artifacts and/or interferences to sample analysis. All these materials must be demonstrated to be free from interferences under the conditions of the analysis by analyzing method blanks.

B. SOLIDS
   1. Sludge samples can contain diverse matrix types, each of which may present its own analytical challenge. Spiked samples and any relevant standard reference material should be processed to aid in determining whether this method is applicable to a given waste.

V. SAFETY

A. Normal accepted laboratory safety practices should be followed while performing this analysis.

B. Be certain the exhaust hood is functioning before you begin the digestion procedure.

C. Hot acids can be extremely corrosive. Avoid inhalation or contact with skin.

VI. EQUIPMENT/APPARATUS

A. Fume hood, Labconco or equivalent.

B. Hot plate, Thermolyne cimarec-3 or equivalent source for use at 95°C. The temperature of the hot plate must be monitored via the use of a temperature blank.

C. Thermometer capable of reading 80 to 120 degrees C – ERTCO cat# 611-3-SC or equivalent.

D. Vacuum pump for filtering dissolved metals- Gast or equivalent.
E. Analytical balance capable of weighing to 0.01 gram. Mettler model BB300 or equivalent.
F. Beckman CS-6R centrifuge.
G. Various class A volumetric glassware and ribbed watchglasses, Pyrex or equivalent.
H. Whatman No. 41 filter paper or equivalent.
I. Whatman No. 42 filter paper or equivalent.
J. Whatman 0.45 micron filter paper or equivalent.
K. 250 mL beaker or other appropriate vessel such as polypropylene block digester tubes, watch glasses and caps.
L. Stirring device, e.g. magnetic stirrer, glass rod or equivalent.
M. Manual Sample Mill
N. Wiley Sample Mill
O. Clippers for cutting vegetation

NOTE: All glassware should be acid washed.

VII. REAGENTS AND STANDARD PREPARATION

A. REAGENTS

1. Metals grade Nitric acid (HNO_3). Reagent should be analyzed to determine level of impurities. If method blank is <MDL, then the reagent can be used.
2. Metals grade Hydrochloric acid (HCl). Reagent should be analyzed to determine level of impurities. If method blank is <MDL, then the reagent can be used.
3. 30% hydrogen peroxide reagent, ACS Grade. Reagent should be analyzed to determine level of impurities. If method blank is <MDL, then the reagent can be used.
4. Metals grade Sulfuric acid (H_2SO_4). Reagent should be analyzed to determine level of impurities. If method blank is <MDL, then the reagent can be used.
5. Reagent water (Deionized water).
6. Potassium Permanganate - Ultra pure grade. Reagent should be analyzed to determine level of impurities. If method blank is <MDL, then the reagent can be used.
7. Ammonium hydroxide, concentrated, reagent grade. Reagent should be analyzed to determine level of impurities. If method blank is <MDL, then the reagent can be used.
8. Ammonium phosphate, reagent grade- Reagent should be analyzed to determine level of impurities. If method blank is <MDL, then the reagent can be used.

B. STANDARDS

1. Traceability

a. A LIMS record shall be maintained on all reference materials. The record shall include date of receipt, source, purity, all compositional information, storage conditions and expiration date. These materials/solutions are to be identified by a unique number in the LIMS as well as on the container's label.
b. All working standards made from reference materials shall be labeled with a unique ID number with complete information on preparation date, concentration of each compound, solvent, preparer's name, expiration date and the information is recorded in LIMS. Reagents shall be labeled with date received and expiration date, if applicable. All of the information described above shall also be recorded in LIMS. Measurements made during standards preparation (e.g., from weighing operations, volume diluted to, etc.) shall also be recorded. There should be no container with sample, sample extract, standard solution, etc. that is not correctly labeled and properly stored.

c. The analyst must initial and date each entry made in a logbook. Each analyst must be sure to "Z" out and initial/date the unused area of each logbook page.

2. PREPARATION

A. Laboratory control sample

1. Aqueous

a. This solution is prepared as follows: 50 mL concentrated HCl, 20 mL concentrated HNO_3, 1 mL of CLP-CAL-1, Solution A, 1 mL of CLP-CAL-1 Solution B, 0.25 mL of CLP-CAL-2, and 0.25 mL of CLP-CAL-3 diluted to 1 L in a volumetric flask. Use 50 mL (100 mL for strict CLPIlM0 4.1) for digestion. This solution is given a unique identifier and recorded in sample digestion logbook/LIMS.

b. For four times concentrated samples: The solution is prepared as follows: 50 mL concentrated HCl, 20 mL concentrated HNO_3, 1mL CLPP-SPK-4 (Inorganic Ventures) (This solution contains 10 mg/L Selenium, 100 mg/L Antimony, 50 mg/L Cadmium and Thallium, 40 mg/L Arsenic and 20 mg/L Lead) to 1 L in a volumetric flask. This solution is given a unique identifier. Use 12.5 mLs to 50 mLs and prepare two aliquots. Heat at 90 to 95°C to reduce the volume in each vessel to ten mLs and then combine each 10 mL aliquot into one vessel and take to a final volume of 25 mLs. Take care to matrix match acids so that the final 25 mL portion will contain 2% HNO_3 and 5% HCl. Use 0.125 mLs HNO_3 and 0.3125 mLs HCl to each 50 mL vessel.

2. Solids:

a. 1.0 ±0.02 (or 2.0 ±0.02) gram aliquot of teflon chips is weighed and spiked using the same spiking solution used for matrix spikes. This sample is given a unique identifier according to the Lot# for the teflon chips used and when digested is given the descriptor. i.e. LCSS(date)A and then B etc. plus the unique identifier number assigned. Alternatively a solid matrix standard reference material is obtained from the manufacturer. This sample is given a unique identifier and the weight is recorded in a bound logbood and transfer to LIMS.

B. Spiking solution

1. Sample is spiked using 0.1 mL of CLP-CAL-1, Solution A, 0.1 mL of CLP-CAL-1 Solution B, 0.025 mL of CLP-CAL-2 and 0.025 mL of CLP-CAL-3 for a final volume of 100 mL. If only 50 mL is used, decrease amount used appropriately. These solutions are given unique identifiers. Record the amount spiked and the unique identifier of the standard.
2. CLP sample is spiked using 0.1 mL CLPP-SPK-1 and 0.1 mL CLPP-SPK-4 for a final volume of 100 mL. If only 50 mL is used, decrease amount used appropriately. These solutions are given unique identifiers.
3. For samples that require four times concentration, the sample is spiked using 0.0125 mLs of CLPP-SPK-4 to each of two vessels with 50 mLs of sample in each. The volume of each of the vessels is lowered to less then 10 mLs and combined and the final volume of this concentrated sample is 25mLs.

VIII. CALIBRATION

A. The temperature of the samples must be maintained at 95°C and monitored via a temperature blank. Record on LIMS batch sheet for later transfer into LIMS.

IX. PROCEDURE

A. Glassware preparation for digestion or when the hot-block can not be used:
   1. Wash glassware with hot soapy water and rinse thoroughly. (Beakers must be washed as soon as possible after being used, dirty beakers must not be allowed to sit overnight.)
   2. Rinse glassware with reagent water that contains 5% HNO_3 and 5% HCl followed by a rinse with reagent water.
   3. Prior to use, all glassware must be confirmed clean via a glassware check. Otherwise, repeat step "2" until the glassware check passes.

B. Aqueous sample filtration (for dissolved metals):
   1. Thoroughly clean a flask and funnel with hot soapy water. Next, rinse the flask and funnel with 1:5 HNO_3 followed by a thorough D.I. water rinsing. This step is very important because the filters contain some metals (namely Zn) which could contaminate the samples.
   2. Rinse a 0.45 micron filter with 1:5 HNO_3 thoroughly, followed by D.I. water.
   3. Filter the unpreserved sample. If dissolved Hg analysis is requested for the sample, filter at least 200 mL.
   4. Discard the first 50 to 100 mL.
   5. A preparation blank must be taken through the filtration step and analyzed with the sample.
   6. Preserve the sample with HNO_3 to pH<2.
   7. Soluble samples that are clean and clear do not have to be digested. Use 100 mL sample, add 5 mL of concentrated HCl and 2 mL of concentrated HNO_3. **Samples must be digested unless approval for analysis without digestion is received from the project manager.**

C. Aqueous sample preparation
   1. Method 3005A and USEPA CLP ILM0 4.1, "**Acid digestion procedure for total recoverable or dissolved metals for analysis by ICP ".**
      a. Shake sample thoroughly and pour 50 mL of the well-mixed sample into a digestion vessel. For samples which require concentration pour 50 mLs of the well-mixed sample into two digestion vessels.
      b. Add 0.50 mL (1 mL of (1+1) when strict CLP ILM0 4.1 is required) concentrated HNO_3 to the sample. For samples which require concentration, add 0.125 mL (0.25 mL of (1+1) when strict CLP ILM0 4.1 is required) concentrated HNO_3 to the sample.
      c. Add 2.5 mL (5 mL of 1+1) when strict CLP ILM0 4.1 is required) concentrated HCl to the sample. For samples which require concentration, add 0.3125 mL (0.625 mL of (1+1) when strict CLP ILM0 4.1 is required) concentrated HCl to the sample.
      d. Cover the sample with a ribbed watch glass or equivalent source.
      e. Transfer the digestion vessel to a pre-heated hot plate or hot block at 90 to 95°C. A temperature blank will assure correct temperature. The temperature must be recorded in the digestion log book. Take the volume down to between 5 to 10 mL, (

12 to 25 mLs when strict CLP ILM0 4.1 is required) **making certain that the sample does not boil**. **This is extremely important. Boiling may lead to vaporization of certain analytes.** Remove the sample from the hot plate and cool

f. When necessary, filter or centrifuge the sample to remove insoluble material that could clog the nebulizer. The filtering apparatus must be thoroughly cleaned with dilute nitric acid prior to filtration.
g. Bring sample to its predigestion volume (or when samples require concentration, to a volume four times lower then what was started with) with DI water in the digestion vessel. The final volume must be recorded in the digestion log book.
h. The sample is now ready for analysis.
i. The digestion log must contain the date, analyst, sample number, client, sample mass/volume, final volume of digestate, lot # of acids used and the preparation and ID of standards plus identification #'s for standards used for spiking and the volume spiked into the sample.

2 Method 200.7, "**Acid digestion procedure for total recoverable metals**".

a. Shake sample thoroughly and pour 50 mL of the well-mixed sample into the digestion vessel. If sample contains undissolved solids >1% refer to Section 11.3 of Method 200.7 for subsequent procedures.
b. Add 1.0 mL concentrated HNO_3 to the sample.
c. Add 2.50 mL concentrated HCl to the sample.
d. Cover the sample with a ribbed watch glass or equivalent source.
e. Transfer the digestion vessel to a pre-heated hot plate or equivalent source at 85°C. Take the volume down to between 10 to 15 mL, **making certain that the sample does not boil**. **This is extremely important. Boiling may lead to vaporization of certain analytes.**
f. Leave sample on hot plate and gently reflux for 30 minutes. Remove from hot plate and cool.
g. Bring sample to its predigestion volume with DI water in the digestion vessel.
h. When necessary, filter or centrifuge the sample to remove insoluble material that could clog the nebulizer. The filtering apparatus must be thoroughly cleaned with dilute nitric acid prior to filtration.
i. The sample is now ready for analysis.
j. The digestion log must contain the date, analyst, sample number, client, sample mass/volume, final volume of digestate, lot # of acids used and the preparation and ID of standards.

3. Method 3010A, "**Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by ICP Spectroscopy**".

a. Shake sample thoroughly and pour 50 mL (5ml diluted to 50mL for TCLP, full 50ml volume for SPLP) of the well-mixed sample into the digestion vessel.
b. Add 1.5 mL concentrated HNO_3 to the sample.
c. Cover the sample with a ribbed watch glass.
d. Transfer the digestion vessel to a pre-heated hot plate or hot block at 90 to 95°C. A temperature blank must be used, with the temperature being recorded in the log book. Take the volume down to a low volume (~5 mL), **making certain that the sample does not boil**. **This is extremely important. Boiling may lead to vaporization of certain analytes. Also make certain that no portion of the**

bottom of the digestion vessel is allowed to go dry. This may lead to low recoveries. Remove the sample from the hot plate and cool.

e. Add another 1.5 mL portion of concentrated HNO_3 to the sample.
f. Cover the sample with a ribbed watch glass.
g. Transfer the vessel to the hotblock or equivalent source. Increase the temperature so a gentle reflux occurs. Continue heating, adding additional acid as necessary, until the digestion is complete (generally indicated when the digestate is light in color or does not change in appearance with continued refluxing).
h. Uncover the vessel and evaporate to a low volume (~3 mL) **making certain that no portion of the bottom of the digestion vessel is allowed to go dry.** Remove and cool.
i. Add 2.5 ml of 1:1 HCl (10 mL/100 mL of final solution).
j. Cover the digestion vessel and reflux for an additional 15 minutes.
k. Bring sample to its predigestion volume in digestion vessel.
l. When necessary, filter or centrifuge the sample to remove insoluble material that could clog the nebulizer. The filtering apparatus must be thoroughly cleaned with dilute nitric acid prior to filtration.

   Note: When preparing DoD project samples, if any sample in a digestion batch requires filtration, all samples (including QC samples) must be treated in the same manner.
m. The sample is now ready for analysis.
n. The digestion log must contain the date, analyst, sample number, client, sample mass/volume, final volume of digestate, lot # of acids used and the preparation and ID of standards.

4 Method 3030C (Standard Methods), "**Preliminary treatment for Acid-Extractable Metals"**
   a. Shake sample thoroughly and pour 50 mL of the well-mixed sample into a 50 mL digestion vessel.
   b. Add 2.5 mL 1:1 HCl to the sample.
   c. Heat 15 minutes in a hot bath.
   d. Filter through a membrane filter.
   e. Adjust filtrate volume to 50 mL with DI water.
   f. Transfer to ICP analyst.

D. Solid sample preparation

It is extremely important that waste (when appropriate), soil and sediment samples be mixed thoroughly to ensure that the sample is as representative as possible of the sample media. The most common method of mixing is referred to as quartering. The quartering procedure should be performed as follows:

- *The material in the sample pan(inorganic-plastic/organic-aluminum) should be divided into quarters and each quarter should be mixed individually.*
- *Two quarters should then be mixed to form halves.*
- *The two halves should be mixed to form a homogenous matrix.*

This procedure should be repeated several times until the sample is adequately mixed.

NOTE: Samples that are clay type materials should be handled in a different manner. Due to these type sample matrices having an affinity to stick to most anything that touches it, another approach must be followed. Obtain a representative sub-sample aliquot from the center or middle section of the sample container.

Grinding of Vegetation Samples

Remove sample from shipping container and brush off dirt particles. Chop sample into about half inch pieces with clippers or other cutting tool. Place the sample in an aluminum pan and air-dry in an exhaust hood to the appropriate dryness for grinding. It should be dry enough where it won't stick to the inside of the mill. Grind the dried sample to fineness in either the manual sample mill or the Wiley mill or both if needed. Place the ground sample in a container and label immediately.

1. USEPA CLP ILM0 4.1, **"Acid digestion of Soil/Sediment"**
   a. Mix the sample thoroughly to achieve homogenity. For each digestion procedure, weigh (to the nearest 0.01 g) a 1.0 to 1.5 g portion of sample and transfer to a digestion vessel.
   b. Add 10 mL of 1:1 nitric acid (HNO_3), mix the slurry, and cover with a watch glass or equivalent source. Heat the sample to 92 to 95°C and reflux for 10 minutes without boiling. Allow the sample to cool, add 5.0 mL of concentrated HNO_3, replace with watch glass or equivalent source, as appropriate, and reflux for 30 minutes. Do not allow the volume to be reduced to less than 5 mL while maintaining a covering of solution over the bottom of the heating vessel.
   c. After the second reflux step has been completed and the sample has cooled, add 2 mL of Type II water and 3.0 mL of 30% hydrogen peroxide (H_2O_2). Return the heating vessel to the hot plate or equivalent heating source for warming to start the peroxide reaction. Care must be taken to ensure that losses do not occur due to excessively vigorous effervescence. Heat until effervescence subsides, and cool the heating vessel.
   d. Continue to add 30% H_2O_2 in 1 mL aliquots with warming until the effervescence is minimal or until the general sample appearance is unchanged. (NOTE: Do not add more than a total of 10 mL 30% H_2O_2.)
   e. If the sample is being prepared for ICP analysis of Al, As, Sb, Ba, Be, Ca, Cd, Cr, Co, Cu, Fe, Pb, Mg, Mn, Ni, K, Se, Ag, Na, Tl, V, and Zn, add 5 mL of 1:1 HCl and 10 mL of Type II water, return the covered heating vessel to the hot plate or equivelent heating source, and heat for an additional 10 minutes. After cooling, filter through Whatman No. 42 filter paper (or equivalent) and dilute to 50 mL with Type II water. NOTE: In place of filtering, the sample (after dilution and mixing) may be centrifuged or allowed to settle by gravity overnight to remove insoluble material. Dilute the digestate to 144 mL with DI water, add 5 mLs concentrated HCl and 1 mL of concentrated $HNO_{3,}$ mix well and place into the appropriate container. The diluted sample has an approximate acid concentration of 2.5% (v/v) HCl and 5% (v/v) HNO_3. The sample is now ready for analysis.
   f. The digestion log must contain the date, analyst, sample number, client, sample mass/volume, final volume of digestate, lot # of acids used and the preparation and ID of standards and ID of matrix spikes and the amounts used for spiking.
2. Method 3050B, **"Acid digestion of Sediments, Sludges and Soils"**

a. Mix the sample thoroughly for 5 minutes using a plastic spatula or Teflon coated spatula in a glass or plastic weigh boat to achieve homogeneity.
b. Weigh approximately (to the nearest 0.01 g) a 1 to 1.5 g portion of the sample directly into a digestion vessel. For samples with low percent solids a larger sample size may be used as long as digestion is completed. Record the exact mass in the digestion log.
NOTE: To achieve the lowest reporting limit possible, use a 2.0 g portion of sample with an ending volume of 100 mLs.
c. Add 5 mL D.I. water and 5 mL concentrated HNO_3(1:1), mix the slurry and cover with a watch glass. Place the sample in a preheated hot block and reflux at 95°C for 10 to 15 minutes being certain that the sample does not boil. Record temperature in digestion log book
d. Allow the sample to cool. Add 5 mL concentrated HNO_3, replace the watch glass and heat/reflux again for 30 minutes. If brown fumes are generated, indicating oxidation of the sample by HNO_3, repeat this step (addition of 5 mL of concentrated HNO_3) over and over until <u>no</u> brown fumes are given off by the sample indicating the complete reaction with HNO_3. Using a watch glass or equivalent allow the solution to evaporate to approximately 5 mL without boiling at 95°C $\pm$ 5°C for approximately two hours. Maintain a covering of solution over the bottom of the vessel at all times. Do not allow the volume to be reduced to less than 5 mL while maintaining a covering of solution over the bottom of the beaker. If the volume does get low, add 2.5 mL of D.I. water to bring volume back up.
e. Take the sample off the hot block and allow it to cool. Next, add 2 mL of D.I. water and 3 mL of 30% Hydrogen Peroxide. (The sample will bubble upon the addition of H_2O_2 if it is still warm.) Cover the vessel with a watch glass and return the sample to the hot block or equivalent source and heat until the bubbling subsides. Care must be taken to ensure that losses do not occur due to excessively vigorous effervescence. Heat until effervescence subsides and cool the beaker. Add two more 3 mL portions of H_2O_2 to the sample in the same manner as before. (NOTE: Do not add more than a total of 10 mL 30% H_2O_2.)
f. Cover the sample with a ribbed watch glass and continue heating the acid-peroxide digestate at 95°C $\pm$ 5°C without boiling for approximately two hours until the volume has been reduced to approximately 2.5 mL. Maintain covering of solution over the bottom of the vessel at all times.
g. Add 2.5 mL of DI water and 2.5 mL of concentrated HCl and 10 mL of DI water, cover the sample with a ribbed watch glass and continue refluxing for an additional 10 minutes without boiling
h. When necessary, filter or centrifuge the sample to remove insoluble material that could clog the nebulizer. The filtering apparatus must be thoroughly cleaned with dilute nitric acid prior to filtration.
i. Bring sample up to 50 mL with D.I. water in the vessel. Add 150 ml of DI water to a 250 ml sample bottle. Invert the 50 ml sample digestion vessel several times to mix the sample and pour sample into the 150 ml of the sample bottle. Pour some sample back into the 50 ml sample digestion vessel to rinse and pour back into the 250 ml sample bottle and cap and mix.

NOTE1: When preparing DoD project samples, if any sample in a digestion batch requires filtration , all samples (including QC samples) must be treated in the same manner.

NOTE2: To achieve the lowest reporting limit possible use 2.0 grams of sample with an ending volume of 100 mLs.

j. The sample is now ready for analysis.
k. The digestion log must contain the date, analyst, sample number, client, sample mass/volume, final volume of digestate, lot # of acids used and the preparation and ID of standards.

X. CALCULATIONS

A. The analyst must be supplied with both beginning sample masses/volumes and final digestate volumes. This information must be recorded in the digestion log.

XI. QUALITY CONTROL

A. Digestion
1. Temperature blank
    a. The temperature of the hot plate/hot block must be monitored for temperature during the digestion process.
    b. The thermometer must be tagged with annual calibration information. Record the thermometer reading, correction factor and the corrected temperature in the digestion log.
2. Blanks
    a. Digest a blank with every batch of samples digested (20 sample maximum). The blank is prepared by adding all the same reagents added to the samples to a clean dry beaker and taking it through the same process as the samples.
    b. Also, there must be a blank for every different method of digestion that is set up that day, every 20 samples.
    c. There must also be a blank for every different matrix of samples that is to be digested, every 20 samples.
    d. Sample is given a unique identifier in the digestion log.
3. Laboratory Control Samples
    a. For water samples, one LCS is digested with every batch of samples digested (20 sample maximum).
    b. For water samples, a LCS is digested every day for each type of digestion, every 20 samples.
    c. For soil/sediment samples, a soil matrix standard reference material (SRM) must be digested per batch (20 samples maximum) or alternatively a spiked teflon chip sample.
    d. Sample is given a unique identifier in the digestion log.
4. Duplicates
    a. A duplicate is prepared every 20 samples. This usually takes the form of a matrix spike duplicate.

    NOTE: Certain projects require a sample duplicate and a matrix spike duplicate with each set of twenty samples.
5. Blank Spike
    a. This is required for certain projects.

B. Sample Matrix

NOTE: Field blanks/duplicates, trip blanks, or equipment blanks are not to be used for sample matrix QC samples.

1. Matrix spike
   a. Digest a spike and spike duplicate every 20 samples where sample volume is adequate to do so. Choose a sample (if possible) that has a lot of metals requested to be analyzed.

      NOTE: For some projects, a sample duplicate and sample spike may be required instead of a spike and spike duplicate. Your supervisor should make you aware of these projects.

   b. The following metals do not get digested spikes when using CLP spike.
      Calcium
      Magnesium
      Sodium
      Potassium
   c. For TCLP samples, a spike must be digested for every matrix. You should inspect the sample (original sample prior to extraction) or check the log book to determine matrix type. (Also the matrix spike aliquot must be added to the extract after filtration but before preservation.)
   d. The CLH project requires that a high and a low spike be prepared and analyzed. Spikes shoud be prepared at 40 mg/Kg and 400 mg/Kg for soil samples and 200 ug/L and 2000 ug/L for aqueous samples.

XII. CORRECTIVE ACTIONS

A. Sample boils during digestion.
   1. Redigest another sample aliquot.

B. Sample goes dry or portion of beaker bottom is exposed due to excess evaporation during digestion.
   1. Redigest another sample aliquot.
   2. Glass beaker dry for an extended period of time? Discard beaker.

XIII. SPECIAL NOTES

A. **Never** take for granted how a sample should be digested. If the sample looks strange or unusual, or if you are not sure what metals the sample gets, what detection limits are required, whether the sample is total or dissolved, or even what method of digestion should be used, always ask your supervisor or the person who is to analyze the sample. How metals need to be digested changes too often to take it for granted.

B. **Antimony (Sb) soils** should be analyzed within 48 hours of digestion whenever possible. When a soil requesting Antimony analysis is received, you must coordinate with the person who will be analyzing it to be sure that they can analyze it on the same day that it is digested.

C. Labels for the digested sample must be written in a neat and legible manner. The labels must include such information as sample number, client name, the date digested, and the volume or mass digested.

D. There are several precautions that must be taken to minimize the possibility of contamination.
   1. All metals glassware must be kept separate from all other laboratory glassware.

2. Metals glassware must be washed as soon as possible after being used. **Dirty metals beakers must not be left overnight.**
3. Acid to be used for metals digestions must be kept separate from all other laboratory acid.

E. Samples must be digested in a timely manner to ensure ICP analysis remains on schedule for data generation. Samples received on or before Wednesday of week X must be prepared for ICP digestion by the end of week X. Your supervisor must be consulted if this schedule can not be met at a particular time.

F. Please consult Waste Disposal SOP-QS14, for information concerning disposal of waste generated from this area. Quantity of chemicals purchased should be based on expected usage during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

Addendum for USEPA CLPILM 05.2 AQUEOUS & SOIL/SEDIMENT

The following is a list of changes for sample preparation when the 5.2 statement of work is required:

1. Soluble samples are required to be digested unless the chain of custody specifically states that digestion is not required. An MDL study must be done on the unprepared MDL solution in order to provide MDL levels for samples that are not digested. When digestion is not required an LCSW and post digestion spike are not required.
2. Digestates must be stored until 365 days after delivery of a complete, reconciled data package.
3. Preparation codes are used on form 13's. They are found in the 5.2 statement of work page B-39 3.4.12.2.4.

DEFINITIONS – Refer to SOP-QS08 for common environmental laboratory definitions.

EMPIRICAL LABORATORIES, LLC
STANDARD OPERATING PROCEDURE

METALS: SOP 105 REVISION #: 16 EFFECTIVE DATE: 041110

METALS
BY INDUCTIVELY COUPLED PLASMA-ATOMIC EMISSION SPECTROMETRY (ICP-AES) TECHNIQUE

References: SW-846, Method 6010B, December 1996; SW-846, Method 6010C, Revision 3 February 2007; USEPA, Method 200.7, June 1991; Standard Methods 19th Edition 2340B; 1995 USEPA CLP, ILM 04.1. See Addendum for USEPA CLPILM 05.2

APPROVALS:

Lab Director: ____________________ Date: 4/12/10

Data Quality Manager: Maria ____________________ Date: 4/11/10

Section Supervisor: Betty DeVille ____________________ Date: 4/13/10

Changes Summary

Revision 16, 04/11/10

- The SOP is an update from Revision 15 dated 05/08/09
- The SOP is formatted to include all 22-elements required per the NELAC standards
- The laboratory's revision of all technical SOPs now includes a Table of Contents that provides the map of the technical information contained within the SOP.
- Additional requirements, based upon the DoD QSM 4.1, have been integrated into the routine sample flow; however, if the requirement is different from routine sample flow, then the requirement is outlined and documented as such to be followed only when DoD samples are analyzed.

Table of Contents

1. Identification of the Test Method

This SOP is compliant with methods – SW846 6010B, SW846 6010C, EPA 200.7, (SM 19th Edition 2340B) Hardness Calculation, (USEPA CLP) ILMO 4.1 (NJDEP does not accept CLPILM 04.1 after June, 2003) and Addendum for USEPA CLPILM 05.2.

2. Applicable Matrix or Matrices

This SOP is applicable to all matrices, including ground water, aqueous samples, TCLP, SPLP and EP extracts, industrial and organic wastes, soils, sludge samples, sediments, and other solid wastes, require digestion prior to analysis.

3. Detection Limit: Detection limits, sensitivity, and optimum ranges of the metals may be found in the ICP method file.

4. Scope of Application, Including components to be Analyzed

Each parameter that is analyzed and reported under the scope of this SOP is listed in **Table 1** of this SOP. This table also lists the associated Method Detection Limit and the Reporting Limit (also defined as the Limit of Quantitation).

5. Summary of the Test Method

5.1 Prior to analysis, samples must be solubilized or digested using appropriate Sample Preparation Methods (e.g., Methods 3005-3050 and SOW ILM 04.1/05.2). When analyzing for dissolved constituents, acid digestion is not always necessary if the samples are filtered and acid preserved prior to analysis. If particulates form after filtration and preservation the sample must be digested prior to analysis.

NOTE: When selenium is required soluble samples must always be digested.

5.2 This method describes the simultaneous multi-elemental determination of elements by ICP. The method measures element-emitted light by optical spectrometry. Samples are nebulized and the large droplets are removed by a spray chamber and the small droplets then pass through to the plasma. The solvent is evaporated. The residual sample decomposed to atoms and ions that become excited and emit characteristic light which is measured, giving a measurement of the concentration of each element type in the original sample. Background correction is required for trace element determination. Background must be measured adjacent to analyte lines on samples during analysis. The position selected for the background-intensity measurement, on either or both sides of the analytical line, will be determined by the complexity of the spectrum adjacent to the analyte line. The position used must be free of spectral interference and reflect the same change in background intensity as occurs at the analytic wavelength measured. Background correction is not required in cases of line broadening where a background correction measurement would actually degrade the analytical result. Control of the spectrometer is provided by PC based *iTEVA* software.

5.3 Inductively Coupled Argon Plasma (ICAP) primary advantage is that it allows simultaneous determination of any elements in a short time. The primary disadvantage of ICP is background radiation from other elements and the plasma gases. Although all ICP instruments utilize high-resolution optics and background correction to minimize these interferences, analysis for traces of metals in the presence of a large excess of a single metal is difficult. Examples would be traces of metals in an alloy or traces of metals in a limed (high calcium) waste. ICP and Flame AA have comparable detection limits (within a factor of 4) except that ICP exhibits greater sensitivity for refractories (Al, Ba, etc.). Furnace AA, in general, will exhibit lower detection limits than either ICP or FAA.

5.4 It is standard procedure to use an internal standard (scandium) with samples to increase the stability of the instrument as recommended by the manufacturer (Thermo Fisher). (When samples are suspected of containing scandium, internal standard cannot be used.)

6. Definitions

Laboratory Quality System SOP QS08 "Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures" provides information on the commonly used definitions.

Additional definitions specific to this SOP are listed below:

6.1 **ICP or ICAP**- Inductively Coupled Plasma or Inductively Coupled Argon Plasma.

6.2 **Inter-element correction (IEC)**- Defined as a correction factor applied by the instrument when there is an overlap of the spectrum from the plasma gases or from another metal into the spectrum of another metal causing that metals concentration to either be inflated or deflated.

7. Interferences

7.1 Spectral interferences are caused by background contribution from continuum or recombination phenomena, stray light from the line emission of high- concentration elements, overlap of a spectral line from another element, or unresolved overlap of molecular band spectra.

7.1.1. Background emission and stray light can usually be compensated for by subtracting the background emission determined by measurements adjacent to the analyte wavelength peak. Spectral scans of samples or single element solutions in the analyte regions may indicate when alternate wavelengths are desirable because of severe spectral interference. These scans will also show whether the most appropriate estimate of the background emission is provided by an interpolation from measurements on both sides of the wavelength peak or by measured emission on only one side. The locations selected for the measurement of background intensity will be determined by the complexity of the spectrum adjacent to the wavelength peak. The locations used for routine measurement must be free of off-line spectral interference (inter-element or molecular) or adequately corrected to reflect the same change in background intensity as occurs at the wavelength peak. For multivariate methods using whole spectral regions, background scans should be included in the correction algorithm. Off-line interferences are handled by including spectra on interfering species in the algorithm.

7.1.2. To determine the appropriate location for off-line background correction, the user must scan the area on either side adjacent to the wavelength and record the apparent emission intensity from all other method analytes. This spectral information must be documented and kept on file. The location selected for background correction must be either free of off-line inter-element spectral interference or a computer routine must be used for automatic correction on all determinations. If a wavelength other than the recommended wavelength is used, the analyst must determine and document both the overlapping and nearby spectral interference effects from all method analytes and common elements and provide for their automatic correction on all analyses. Tests to determine spectral interference must be done using analyte concentrations that will adequately describe the interference. Normally, 100 mg/L single element solutions are sufficient; however, for analytes such as iron that may be found at high concentration, a more appropriate test would be to use a 200 mg/L or 500 mg/L concentration near the upper analytical range limit.

7.1.3. Spectral overlaps may be avoided by using an alternate wavelength or can be compensated by equations that correct for inter-element contributions. Instruments that use equations for inter-element correction require the interfering elements be analyzed at the same time as the element of interest. When operative and uncorrected, interferences will produce false positive determinations and be reported as analyte concentrations. More extensive information on interferant effects at various wavelengths and resolutions is available in reference wavelength tables and books. Users may apply inter-element correction equations determined on their instruments with tested concentration ranges to compensate (off line or on line) for the effects of interfering elements. Some potential spectral interferences observed for the recommended wavelength are listed in the method in table 2. For multivariate methods using whole spectral regions, spectral interferences are handled by including spectra of the interfering elements in the algorithm. The interferences listed are only those that occur between method analytes. Only interferences of a direct overlap nature are listed. These overlaps were observed with a single instrument having a working resolution of 0.035 nm.

7.1.4. When using inter-element correction equations, the interference may be expressed as analyte concentration equivalents (i.e. false analyte concentrations) arising from 100 mg/L of the interference element. For example, assume that Arsenic is to be determined (at 193.696 nm) in a sample containing approximately 10 mg/L of Aluminum. According to Table 2 from the method, 100 mg/L of Aluminum would yield a false signal for Arsenic equivalent to approximately 1.3 mg/L. Therefore, the presence of 10 mg/L of Aluminum would result in a false signal for Arsenic equivalent to approximately 0.13 mg/L. The user is cautioned that other instruments may exhibit somewhat different levels of interferences than that shown in Table 2 from the method. The interference effects must be evaluated for each individual instrument since the intensities will vary.

7.1.5. Inter-element corrections will vary for the same emission line among instruments because of differences in resolution, as determined by the grating, the entrance and exit slit widths, and by the order of dispersion. Inter-element corrections will also vary depending upon the choice of background correction points. Selecting a background correction point where an interfering emission line may appear should be avoided when practical. Inter-element corrections that constitute a major portion of an emission signal may not yield accurate data. Users should not forget that some samples may contain uncommon elements that could contribute spectral interferences.

7.1.6. The interference effects must be evaluated for each individual instrument. For each instrument, intensities will vary not only with optical resolution but also with operating conditions (such as power, viewing height and argon flow rate). When using the recommended wavelengths, the analyst is required to determine and document for each wavelength the effect from referenced interferences as well as any other suspected interferences that may be specific to the instrument or matrix. The instrument utilizes a computer routine for automatic correction on all analyses.

7.1.7. If the correction routine is operating properly, the determined, apparent analyte(s) concentration from analysis of each interference solution should fall within a specific concentration range around the calibration blank. The concentration range is calculated by multiplying the concentration of the interfering element by the value of the correction factor being tested and divided by 10. If after the subtraction of the calibration blank the apparent analyte concentration falls outside of this range in either a positive or negative direction, a change in the correction factor of more than 10% should be suspected. The cause of the change should be determined and corrected and the correction factor updated. The interference check solutions should be analyzed more than once to confirm a change has occurred. Adequate rinse time between solutions and before analysis of the calibration blank will assist in the confirmation.

7.1.8 When inter-element corrections are applied, their accuracy should be verified, daily, by analyzing spectral interference check solutions (IFA/IFB). If the correction factors or multivariate correction matrices tested on a daily basis are found to be within 20% criteria for 5 consecutive days, the required verification frequency of those factors in compliance may be extended to a weekly basis. Also, if the nature of the samples analyzed is such they do not contain concentrations of the interfering elements at ± one reporting limit from zero, daily verification is not required. All inter-element spectral correction factors or multivariate correction matrices must be verified and updated every six months or when an instrumentation-change, such as in the torch, nebulizer, injector, or plasma conditions occurs.

Standard solution should be inspected to ensure that there is no contamination that may be perceived as a spectral interference.

7.2. Physical interferences are effects associated with the sample nebulization and transport processes. Changes in viscosity and surface tension can cause significant inaccuracies, especially in samples containing high dissolved solids or high acid concentrations. If physical interferences are present, they must be reduced by diluting the sample or by using a peristaltic pump, by using an internal standard or by using a high solids nebulizer. Another problem that can occur with high dissolved solids is salt buildup at the tip of the nebulizer, affecting aerosol flow rate and causing instrumental drift. The problem can be controlled by wetting the argon prior to nebulization, using a tip washer, using a high solids nebulizer or diluting the sample. Also it has been reported that better control of the argon flow rate, especially to the nebulizer, improves instrument performance: this may be accomplished with the use of mass flow controllers.

7.3. Memory interferences result when analytes in a previous sample contribute to the signals measured in a new sample. Memory effects can result from sample deposition on the uptake tubing to the nebulizer and from the build-up of sample material in the plasma torch and spray chamber. The site where these effects occur is dependent on the elements and can be minimized by flushing the system with a rinse blank between samples. The possibility of memory interferences should be recognized

7.4 Users are advised that high salt concentrations can cause analyte signal suppressions and confuse interference tests. When the instrument displays negative values, dilution of the samples may be necessary.

8. Safety

Laboratory SOP QS13 “Safety Program & Chemical Hygiene Plan” discusses the safety program that is to be followed lab-wide.

8.1 Normal accepted laboratory safety practices should be followed while performing this analysis.

8.1.1. Care should be used in handling all samples. Safety glasses must be worn in the lab at all times. The use of appropriate safety gloves and lab coats is highly recommended.

8.1.2 Research into expected sample content and concentration should be done in order to be prepared for additional safety considerations. Generally, any samples that need special consideration have applicable notes on the sample logs.

8.1.3 MSDS sheets are available for all reagents and standards that have been purchased. These are located in the bookshelves in the Quality Assurance Officers office.

9. Equipment & Supplies

9.1. Inductively coupled argon plasma emission spectrometer: Thermo Scientific 6500 DUO.

9.2. Computer-controlled emission spectrometer with background correction: Thermo Scientific 6500 DUO or equivalent.

9.3. Radio frequency generator compliant with FCC regulations: Thermo Fisher or equivalent.

9.4. Auto-sampler: Thermo Fisher or equivalent.

9.5. Printer capable of printing results every 4 minutes.

9.6. Cooling Water recycler.

9.7. *Iteva* software.

9.8. Argon gas supply – Liquid Argon

9.9. Class A volumetric flasks

9.10. Analytical balance - capable of accurate measurement to a minimum of three significant figures (0.001gm).

9.11. Variable Eppendorf Pipettes 1000µL; 5000µL

9.12. Disposable beakers 10, 20 and 50 mL size.

9.13. Hood system capable of venting the heat from the system off of the instrument during analysis.

10. Reagents and Standards

The laboratory's LIMS system allows for complete documentation and for the traceability of reagents and standards used within the laboratory. The following information relates to the specific reagents and standards used for the performance of the method:

10.1. Reagent Water. All references to water in the method refer to reagent grade water unless otherwise specified. Reagent water will be interference free.

10.2. Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination. If the purity of a reagent is in question analyze for contamination. If the concentration is less than the MDL then the reagent is acceptable.

10.3. Hydrochloric acid (concentrated), HCl. A method blank is digested and analyzed before a new lot number of HCl is put into use, to ascertain purity. The lot # is logged into Element and the data kept on file.

10.4. Nitric acid (concentrated), HNO_3. A method blank is digested and analyzed before a new lot number of HNO3 is put into use, to ascertain purity. The lot # is logged into Element and the data kept on file.

10.5. Calibration standards

10.5.1. All standards have an acid matrix of 2% HNO_3 and 5% HCl and should be prepared using class A volumetric flasks and calibrated Eppendorfs).

10.5.2. CAL1 is the calibration blank: Reagent grade water **matrix matched as in 10.5.1. Note: when this standard is analyzed the intensities should be compared to a previous run to make sure that no contamination has occurred. Prepare this solution fresh daily.**

10.5.3. Stock QC21 solution: (100 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element and includes the following metals - Sb, As, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Li, Mg, Mn, Mo, Ni, Se, Sr, Tl, Ti, V, and Zn.

10.5.4. Stock QC7 solution: Order from the manufacturer already prepared. This solution is given a unique identifier within Element and includes the following metals- (50 ug/mL)- silver; (100 ug/mL)- aluminum, boron, barium and sodium; (1000 ug/mL)- potassium; (500 ug/mL or 100 ug/mL note we use two sources of this standard and each have different concentrations for Si) –Silica.

10.5.5. Boron solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.

10.5.6. Stock Tin solution: (10000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element. Note: Two sources are needed.

10.5.7. Stock Silver solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.

10.5.8. Stock Aluminum solution: (10000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element. Note: Two sources are needed.

10.5.9. Stock Calcium solution: (10000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier. Note: Two sources are needed.

10.5.10. Stock Magnesium solution: (10000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element. Note: Two sources are needed.

10.5.11. Stock Iron solution: (10000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element. Note: Two sources are needed.

10.5.12. Stock Potassium solution: (10000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element. Note: Two sources are needed.

10.5.13. Stock Barium solution: (10000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.

10.5.14. Stock Sodium solution: (10000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element. Note: Two sources are needed.

10.5.15. Stock Arsenic solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.

10.5.16. Stock Cobalt solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.

10.5.17. Stock Chromium solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.

10.5.18. Stock Copper solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.

10.5.19. Stock Manganese solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.

10.5.20. Stock Nickel solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.

10.5.21. Stock Lead solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.

10.5.22. Stock Selenium solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.

10.5.23. Stock Thallium solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.

10.5.24. Stock Beryllium solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.

10.5.25. Stock Cadmium solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.

10.5.26. Stock Antimony solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.

10.5.27. Stock Molybdenum solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.

10.5.28. Stock Strontium solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.

10.5.29. Stock Titanium solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.

10.5.30. Stock Vanadium solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.

10.5.31. Stock Zinc solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.

10.5.32. Stock Scandium solution (10000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.

10.6. Calibration and Calibration Verification standards

10.6.1. The calibration standards and calibration verification standards preparations are recorded in Element. Please find method of preparation in Appendix I.

10.6.2. The CRL solution is analyzed to check the accuracy of the instrument at the reporting limit. The stock standard solutions A and B are prepared from single element standards listed in 10.5 above. Please find method of preparation in Appendix I. This solution is stable for 6 months. The working solutions are made up as needed or every 3 months as follows: Prepared by adding 1.0 ml of RL Stock solution A and 1.0 ml of RL Stock Solution B to de-ionized water with 2% HNO3 and 5% HCL matrix and diluting to 100 mLs , mix well. This solution is stable for 3 months.

10.6.3. The interference check standard solutions (IFA and IFB) are prepared to provide an adequate test of the IECs. A purchased solution containing 500 ug/mL Al, Ca, Mg and 200 ug/mL Fe is diluted 10x to prepare the IFA. The IFB is prepared by diluting 100x a purchased solution containing 10 ug/mL of As and Tl; 20 ug/mL Ag; 50 ug/mL Ba, Be, Cr, Co, Cu, Mn, and V; 100 ug/mL Cd, Ni and Zn; 5 ug/mL Pb and Se; and 60 ug/L Sb. Add to this a purchased solution containing 500 ug/mL Al, Ca, Mg and 200 ug/mL Fe diluted 10x. These solutions are prepared as needed or monthly and assigned an Element # for traceability.

10.7 Digestion standards

10.7.1 The Blank Spike (BS) is prepared from High Purity solutions CLP-CAL-1 solution A and B; CLP-CAL-2 and CLP-CAL-3. 0.50 mL of CLP-CAL-1 A and B; and 0.50 mLs of the 1000 ug/mL single element standards for Molybdenum, Boron, Titanium and Strontium is diluted to 500 mL with 0.125 mL of CLP-CAL-2 and CLP-CAL-3 and 0.050 mLs of 10000 ug/mL Tin. 25 mL of HCl and 10 mL of HNO3 are added for preservation. This solution is stored in a Teflon bottle. A portion is reserved in case of a problem with digestion. When there is a problem with the analysis of the BS the solution is checked first before action is taken to make sure that it was made properly and has not deteriorated since it was made up. This solution is given a unique identifier within Element. The BS is prepared from a source independent from that used in the calibration standards. This solution is prepared daily or as needed. 50 mLs of this solution is used for digestion for normal level water samples and the sample is brought back to 50 mLs after digestion. Low level water samples start with two 50 mLs vials with only 1.0 mL of the stock blank spike solution in each taken to 50 mLs. The samples are cooked down to below 25 mLs and combined and then cooked down to below 25 mLs again and then brought back to 25 mLs. This low level BS is given a unique identifier in Element.

10.7.2. The solid BS used with soil samples is prepared by weighing up 1.0 gram of Teflon chips for regular level and 2.0 grams of Teflon chips for low level and spiking using the same spiking solutions used to spike the sample matrix. This standard is given a unique identifier i.e. Batch #-BS1. Note: Amount of spiking solution used varies according to whether the samples are being digested for normal level or low level soils. See spiking solutions in 10.7.3.1 for how to prepare the BS for a solid sample, it is prepared the same way that a soil spike is prepared only the known amounts of metals are added to laboratory water.

10.7.3. The spiking solutions are prepared as follows:

10.7.3.1.Stock Multi-element Spiking Solutions: High Purity CLP-CAL-1 solution A: 2000 ug/mL Al and Ba; 50 ug/mL Be; 200 ug/mL Cr; 500 ug/mL Co, Mn, Ni, V and Zn; 250 ug/mL Cu; 1000 ug/mL Fe; 5000 ug/mL Ca, Mg, K and Na; solution B: 250 ug/mL Ag; CLP-CAL-2: 1000 ug/L Sb; CLP-CAL-3: 1000 ug/mL As, Pb, Se, Tl; 500 ug/mL Cd. Order from the manufacturer already prepared. These solutions are given a unique identifier within Element. Add 0.050 mL for water samples and 0.20 mL for normal level soil samples and 0.10 for low level soil samples of CLP-CAL-1 solutions A and B, and 0.0125 mL for water samples and 0.05 mLs for normal level soil samples and 0.025 mLs for low level soil samples of CLP-CAL-2 and 3 to 50 mL of sample for water samples and 1gram of sample for normal level soils and 2 grams of sample for low level soils for the following spike values: 2000 ug/L Al and Ba; 50 ug/L Be; 200 ug/L Cr; 500 ug/L Co, Mn, Ni, V and Zn; 250 ug/L Cu; 1000 ug/L Fe; 5.0 mg/L Ca, Mg, K and Na, 250 ug/L Ag, Sb, As, Pb, Se and Tl; 125 ug/L Cd. A blank spike should be prepared at the time the samples are spiked to check the actual spike value and accuracy.

10.7.3.2. <u>TCLP Spiking Solution: Use 0.50 mL diluted to 50 mL for digestion:</u>
2.5 mL 10000 mg/L Ba stock standard diluted to 100 mL; 2.5 mL Cr, Pb and As 1000 mg/L stock standard diluted to 100 mL; 0.50 mL Cd and Se diluted to 100 mL . Store in a Teflon bottle. A blank spike should always be prepared at the same time a sample is being spiked. This solution should produce a spike value of 2500 ug/L Ba; 250 ug/L Cr, Pb and As; and 50 ug/L of Cd and Se. Note: Since the samples are diluted 10x when digested the spike value will appear to be 10x greater when analyzed.

10.7.3.3. <u>TCLP Silver Spiking Solution: Use 5.0 mL diluted to 50 mL for digestion:</u>
0.40 mL of 1000 mg/L stock Ag solution diluted to 200 mL. Store this solution in a Teflon bottle. A blank spike should always be prepared at the same time a sample is being spiked. This solution should produce a spike value of 200 ug/L. Note: Since the samples are diluted 10x when digested the spike value will appear to be 10x greater when analyzed. Also this solution is not very stable and may require fresh preparation at least weekly.

11. Sample Collection, Preservation, Shipment, and Storage

Quality Systems SOP QS10 related to Sample Receipt, Handling, & Processing provides details for collection, preservation, shipment, and storage.

11.1. Preliminary treatment of most matrices is necessary because of the complexity and variability of sample matrices. Water samples which have been pre-filtered and acidified will not need acid digestion as long as the samples and standards are matrix matched and particulates do not form after the filtration and preservation take place. Solubilization and digestion procedures are presented in Sample Preparation Methods (Methods 3005A-3050A).

11.2. Sample digestates are stored at room temperature for at least 2 months unless a longer time is requested by the client. The samples contain an acid matrix of 3:1. All metal samples are neutralized before disposal in the receiving section of the laboratory.

11.3. **The appropriate SOPs should be consulted regarding sample preparation.** The following is a brief summary of the methods we use for metals preparation.

11.3.1. Method 3005A prepares groundwater and surface water samples for total recoverable and dissolved metals determination by ICP. The unfiltered or filtered sample is heated with dilute HCl and HNO_3 prior to metal determination.

11.3.2. Method 3010A prepares waste samples for total metal determination by ICP. The samples are vigorously digested with a mixture of nitric acid and hydrochloric acid followed by dilution with laboratory water. The method is applicable to aqueous samples, TCLP and mobility-procedure extracts.

11.3.3. Standard Methods 19th Edition Method 3030C prepares ground-waters and surface water samples for acid extractable metals: (lead and chromium.) This preparation has a holding time of 72 hours. The samples are preserved at collection with 5mL/L of HNO3, in the laboratory 5 mL/100mL of 1+1 HCl is added and the sample is heated for 15 minutes in a block digester. The sample is filtered through a membrane filter and the filtrate is carefully transferred to a volumetric flask and brought back to 100 mLs.

11.3.4. Method 3050B prepares wastes samples for total metals determination by ICP. The samples are vigorously digested in nitric acid and hydrogen peroxide followed by dilution with either laboratory water or hydrochloric acid and laboratory water. The method is applicable to soils, sludges, and solid waste samples.

12. Quality Control

Quality Systems SOP QS08 "Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures" outlines details related to laboratory wide protocols on quality control.

12.1. **Daily run and batch QC**

12.1.1. Calibration is required daily. Either a blank and a high standard or a client specific three standard concentration points and a blank calibration is required daily.

12.1.2. IEC correction standards for aluminum and iron are required daily.

12.1.3. ICV within ±5% for 200.7 and within ±10% for all other methods.

12.1.4. ICB/CCB less than two times ± MDL or less than ± LOD for DOD. The ICB/CCB must immediately follow the ICV/CCV.

12.1.5. RL standard run against the curve within ±20% initially and client specific requirement of ±30% at the end of the analysis.

12.1.6. IFA/IFB analyzed daily. IFA must be less than two times ±MDL or less than ±LOD unless verified standard contamination for DOD. The IFB must recover within ±20% for all analytes in the IFB standard solution. If the IFA/IFB solution is not within the required limits- if possible reanalyze all associated samples, if not possible to reanalyze all associated samples must be flagged with an "Q" on the final report for DOD.

12.1.7. CCV must be analyzed every ten samples or at the end of the analysis within ±10% or the samples are reanalyzed if possible. If samples cannot be reanalyzed, all samples are flagged with a "Q" for DOD.

12.1.8. CCB must be analyzed every ten samples immediately following the CCV or at the end of the analysis less than two times ±MDL or <±LOD for DOD. If the CCB is out of the allowable range the samples are flagged with "B".

12.1.9. *The following should be analyzed with each preparation batch containing a matrix spike.*

- Serial dilution: If the analyte concentration is sufficiently high (minimally, a factor of 50 above the instrumental detection limit after dilution), an analysis of a 1:4 dilution (volumetric glassware must be used) should agree within ±10% of the original determination. If not, a chemical or physical interference effect should be suspected. The analyst and or section manager must note this situation on the final analytical report.

- Post digestion spike addition: An analyte spike added to a portion of a prepared sample, or its dilution, should be recovered to within 75% to 125% of the known value for SW6010B and 80 to 120% for SW6010C and is required especially if the pre-digestion matrix spike is outside of control limits. The spike addition should produce a minimum level of 10 times and a maximum of 100 times the instrumental detection limit. If the spike is not recovered within the specified limits, a matrix effect should be suspected. Run all associated samples in the preparatory batch by method of standard additions (MSA) or apply "J" flag. The analyst and or section manager must note this situation on the final analytical report. Apply "J" flag if the post spike is outside the range of 75 to 125% for 6010B or 80 to 120% for 6010C.

12.2 Quarterly and/or every six months

12.2.1. Linear range standards must be analyzed at a frequency no less than once every six months. The linear range standard is required for verification that samples are actually linear to the degree claimed. The analyst is responsible for completing this task in a timely manner. The linear range standard must be within +/-10% of true value. This standard can be analyzed as the linear dynamic range.

12.2.2. The inter-element correction factors (IEC) should be verified at the time the linear range standards are analyzed or whenever there is any question about whether an IEC is correcting correctly.

12.2.3. IDL's, linear range and IEC checks must be performed quarterly if straight CLP work is required.

12.3. Digested Batch QC

12.3.1. All quality control data should be maintained and available for easy reference or inspection.

12.3.2. Employ a minimum of one method blank per sample batch to determine if contamination or any memory effects are occurring. A method blank (BLK), sometimes referred to as the preparation blank is a volume of reagent water acidified with the same amounts of acids as were the standards and samples. These blanks are taken through the same digestion/preparation steps as the sample being tested. The result for the method blank should not indicate contamination greater than ± ½ RL for DOD or ±RL/CRDL for other or CLP. If exceeded, the impact upon the data should be evaluated and the associated sample(s) should be either re-digested or the data should be qualified. The extracted blank associated with TCLP batches must be less than 100 X the regulatory limit for barium.

12.3.3. Employ a minimum of one blank spike (BS) for aqueous samples or one Teflon chip spiked sample per sample batch to verify the digestion procedure. These blank spikes are taken through the same digestion/preparation steps as the sample being tested. The control limits are ±15% method 200.7 - aqueous and soil samples or ±20% for all other methods aqueous and soil samples. If the BS is not in control, the impact upon the client data should be evaluated and the associated sample(s) should be re-digested. Consult your supervisor for further action. Qualifying the associated data may not be permissible for some clients.

12.4. Sample

12.4.1. Analyze one replicate sample for every twenty samples or per analytical batch, whichever is more frequent. A replicate sample is a sample brought through the whole sample preparation and analytical process in duplicate. It is acceptable to substitute a matrix spike duplicate for the sample replicate. Project specific requirements will take precedence in these situations. NJDEP demands that this requirement be met with a client specific duplicate rather than a spike duplicate. The control limits are less than or equal to 20% RPD (if both are >5x RL) or ± the RL (if either are <5X RL). Supervisor must be notified if the control limit is not met. Supervisor will dictate corrective action if required. The final analytical report must document this situation. Apply "J" flag for DOD if acceptance criteria are not met. Apply "*" flag for CLP and other work if acceptance criteria are not met.

12.4.2. Analyze a minimum of one spiked sample and/or spiked sample duplicate for every twenty samples or per analytical batch, whichever is more frequent. Project specific requirements will take precedence in determining whether a matrix spike duplicate is employed in these situations. If the analyte level in the sample is not greater than 4X the spiking level, the spike recoveries should be within ±20% of the true value. If not, and sufficient sample volume exist, a post digestion spike should be analyzed. Apply "J" flag for DOD if acceptance criteria are not met. Apply "N" flag or CLP and other work if acceptance criteria are not met.

13. Calibration and Standardization

Quality Systems **SOP QS08 "**Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures" related to Calibration Procedures provides laboratory wide protocols for calibration and standardization.

13.1. Set up the instrument with proper operating parameters. The instrument must be allowed to become thermally stable before beginning (usually requiring at least 30 minutes of operation prior to calibration).

13.2. Operating conditions - **The instrument settings can be found in method file within the iTEVA software.** For operation with organic solvents, use of the auxiliary argon inlet is recommended, as are solvent-resistant tubing, increased plasma (coolant) argon flow, decreased nebulizer flow, and increased RF power to obtain stable operation and precise measurements. Sensitivity, instrumental detection limit, precision, linear dynamic range, and interference effects must be established for each individual analyte line on that particular instrument. The analyst must (1) verify that the instrument configuration and operating conditions satisfy the analytical requirements and (2) maintain quality control data confirming instrument performance and analytical results.

13.3. Auto-peak when some change has been made to the introductory system and calibrate the instrument according to the instrument manufacturers recommended procedures, using the specified calibration standard solutions. Flush the system with 2% HNO_3 / 5% HCl between each standard or as the manufacturer recommends. (Use the average intensity of multiple exposures for both standardization and sample analysis to reduce random error.) The calibration curve consists of a blank and three standards ($r \geq 0.998$). If a three point calibration curve is not required for the client samples being analyzed by Empirical Laboratories may use a blank and one standard as referenced in USEPA - CLP protocols.

13.4. Before beginning the sample run, analyze single element Iron and Aluminum standards at their linear range to check for IEC drifts. Analyze these standards first as QC samples with an IEC check table and action taken should be to calculate IECs using the iTEVA software. Make sure to rinse thoroughly after running these linear range standards, they can cause carry over into the initial QC samples which are analyzed next. The analysis order follows as: ICV (± 10%) for 200.7 (± 5%) and ICB (< ±2xMDL, <±LOD-DOD or ±RL/CRDL for others or CLP, first, then analyze a reporting limit standard (a standard at the concentration of the reporting limit). This standard should be within ±20% for DOD projects and ±30% for samples analyzed for 6010C. Then reanalyze the highest mixed calibration standard(s) as if it were a sample. Concentration values obtained should not deviate from the actual values by more than 5%. If they do, follow the recommendations of the instrument manufacturer to correct for this condition. Note: Supervisor must be notified if the control limit is not met. Supervisor will dictate corrective action if required. The final analytical report must document this situation.

13.5. For **CLP projects**, verify the validity of the curve in the region of 2x the contract required detection limit (CRDL) before and after each batch of 20 samples in the specific order of CRI, ICSA, ICSAB, CCV and CCB (CCB criteria: < ±MDL or ±RL/CRDL for others or CLP, or twice during every 8-hour work shift, whichever is more frequent. Results should be within ±20%. Supervisor must be notified if the control limit is not met. Supervisor will dictate corrective action if required. The final analytical report must document this situation. (For Internal QC)

13.6. Verify the inter-element and background correction factors at the beginning of the sequence in the specific order of IFA, IFB, CCV and CCB (IFA criteria: non-spiked analytes < ±2xMDL or <±LOD for DOD beginning of sequence**.** Do this by analyzing the interference check solution IFA and IFB. Absolute value of concentration for all non-spiked analytes in the IFA must be <LOD (unless they are verified trace impurity from one of the spiked analytes) for DOD. Results must be within ±20% of the true value for IFB. If corrective action fails, apply Q-flag to all results for specific analyte(s) in all samples associated with the ICS. (CRI, ICSA and ICSAB required at the end for CLP projects only).

Note: Supervisor must be notified if the control limit is not met. Supervisor will dictate corrective action if required. The final analytical report must document this situation.

13.7. The instrument must be calibrated once every 24 hours.

13.8. Instrument Autosampler Report example:

Calibration Rack (used by instrument software to insert QC)

1) Cal Std 1 (blank)
2) Cal Std 2 (Low Cal)
3) Cal Std 3 (Mid Cal)
4) Cal Std 4 (Ba @ 5000 ppb)
5) Cal Std 5 (QC5)
6) Cal Std 6 (QC 21)
7) Cal Std 7 (NAK 100)
8) Cal Std 8 (QC3)
9) Cal Std 9 (Ag)
10) Al IEC-(correction using ITEVA software)
11) Fe IEC-(correction using ITEVA software)

Sample Sequence RACK 1

1)	SEQ-ICV
2)	SEQ-ICB
3)	SEQ-CRL1-reporting limit standard 1
4)	SEQ-CRL2-reporting limit standard 2
5)	Ba@ 5000 ppb (readback)
6)	QC5
7)	NAK High-(readback)
8)	QC 21 High-(readback)
9)	Salt Cal at 500 ppm (readback)
10)	Rinse
11)	SEQ-IFA1
12)	SEQ-IFB1
13)	Rinse
14)	SEQ-CCV
15)	SEQ-CCB
16)	Method Blank (*Batch #* -BLK1)
17)	Blank Spike (*Batch #* -BS1)
18)	Sample 1
19)	Sample 2
20)	Sample 3
21)	Sample 4
22)	Sample 5
23)	Sample 6
24)	Sample 7
25)	Sample 8
26)	Sample 9
27)	Sample 10
28)	SEQ-CCV
29)	SEQ-CCB
30)	Sample 11
31)	Sample 12
32)	Sample 13
33)	Sample 14
34)	Sample 15
35)	Sample 16
36)	Sample 17
37)	Sample 18
38)	Sample 19
39)	Sample 20
40)	Sample matrix spike (*batch#*- MS1)
41)	Sample matrix spike duplicate (*batch#* -MSD1)
42)	Sample post digestion spike (*batch#* -PS1)
43)	Sample serial dilution (*batch#* -DUP1)
44)	SEQ-CCV

45) SEQ-CCB
46) Preparation Blank (*batch#* -BLK1)
47) Blank Spike (*batch#* -BS1)
48) Sample 1
49) Sample 2
50) Sample 3
51) Sample 4
52) Sample 5
53) Sample 6
54) Sample 7
55) Sample 8
56) Sample 9
57) Sample10
58) SEQ-CCV
59) SEQ-CCB
60) Sample 11

RACK 2

1) Sample 12
2) Sample 13

Etcetera…

Each rack holds 60 samples and there are 4 racks that are used for samples, CCVs and CCBs and run QC.

14. Procedure

14.1. Once the instrument has been calibrated, begin the analysis of samples.

14.2. If particulates are visible in the digestate, the sample must be filtered prior to analysis. If filtration is required, a filter blank must be prepared by filtering reagent grade water which has been properly acidified. **In the event USACE samples are filtered, all USACE samples and the QC samples in that QC batch must be filtered. All USACE solid samples and their associated batch QC samples must be filtered prior to analysis.**

14.3. Flush the system with 2% HNO_3 / 5% HCl for at least 1 minute before the analysis of each sample.

14.4. Dilute and reanalyze samples that are more concentrated than the linear calibration limit or, for 200.7, ± 10% of the linear range standard. **In the case of USACE samples, the criterion changes and requires dilution and reanalysis of all samples which produce a concentration that exceeds the highest calibration standard. Sample results detected between the MDL and LOQ are flagged as estimated with a "J" flag.**

14.5. Verify calibration every 10 samples or every 2 hours, whichever is more frequent and at the end of the analytical run, using a continuing calibration verification (CCV) sample and a continuing calibration blank (CCB) sample.

14.5.1. The results of the CCV are to agree within ±10% for 6010 (5% for 200.7) on initial verification of the expected value, with relative standard deviation (RSD) < 5% from 3 replicates (minimum of three integrations). If not, terminate the analysis, correct the problem, and reanalyze the previous ten samples. The analyst may continue the analytical run, and after conferring with the section manager it may be necessary to reanalyze a group of samples. The analyst must notify the section manager within 24 hours.

14.5.2. The results of the calibration blank (this is not the method/preparation blank) are to be < 2x ±MDL, for CLP <RL, for **DOD no analytes detected >±LOD**. If the calibration blank is not in control, evaluate the impact upon the previous 10 samples. Reanalysis may be required after an evaluation of the data. If the blank < 1/10 the concentration of the action level of interest and no sample is within 10% of the action limit, samples need not be reanalyzed. One must also evaluate the reporting limit (RL) as it relates to 3X the IDL/MDL. If the RL is significantly above 3X IDL or MDL then reanalysis may not be required (Na, K, Mg and Ca are good examples of this situation).

14.6. Demonstration of Capability (DOC) – Each analyst must perform a DOC to demonstrate proficiency with this method. Refer to SOP-413 for guidance.

15. Data Analysis and Calculations

Quality Systems SOP QS09 “General and commonly used Laboratory Calculations” provides details on general calculations used throughout the laboratory.

15.1. Total hardness is reported from HNO_3 preserved sample. The final concentration is calculated from the calcium and magnesium results as follows: Ca mg/L x 2.5 + Mg mg/L x 4.1 = total Hardness in mg/L as $CaCO_3$.

15.2. The instrument will generate data results in mg/L or µg/L (labeled appropriately). Each result represents an average of three individual readings per metal channel.

15.3. For aqueous samples, if a post/pre-digestion dilution is performed, the result must be multiplied by this factor or the dilution factor must be entered into the instrument data table in which case the instrument will generate data corrected for the dilution.

15.4. For solid samples, if a post-digestion dilution is performed, the result must be multiplied by this factor or the dilution factor must be entered into the instrument data table in which case the instrument will generate data corrected for the dilution. Also, the result must be converted to reporting units which are usually mg/kg.

SR (ug/g or mg/kg) = IR*DF*FED/SM

SR	=	Sample result
IR	=	Instrument result (µg/L)
DF	=	Dilution factor (post digestion)
FED	=	Final volume of digestate (L)
SM	=	Sample mass digested (g)

16. Method Performance

Demonstration of Capability (DOC): Each analyst must perform a DOC prior to reporting data. The analyst must prepare (for prep technicians) and analyze (analysts reviewing and reporting data) 4-LCS samples. The data is calculated for accuracy and precision requirements. The DOC form, as listed within section 2.5 of the Quality Manuel is completed by each analyst and then provided to the supervisor for further processing and approval.
DOC LCS Preparation: See BS preparation under 10.7.1 through10.7.3 above.
DOC Accuracy and Precision Criteria: The LOD is analyzed at 2 times the MDL and must result in an concentration 3 times the noise. The LOQ is analyzed at the RL or 2 times the RL and must be recovered within ±50%.

17. Pollution Prevention:
Quantity of chemicals purchased should be based on expected usage during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

18. Data Assessment and Acceptance Criteria for Quality Control Measures

Quality Control SOP QS05, "Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results", provides details on data assessment and acceptance criteria for Quality Control Measures. Table 2 of this SOP provides information on QC samples, frequency, and the associated criteria specific to the performance of this method.

19. Contingencies for Handling out-of-control or unacceptable data
Quality Control SOP QS05, "Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results", provides details on handling out of control data. Table 2 within this SOP also lists corrective actions associated with the failure of the various QC samples employed for the performance of this method.

CORRECTIVE ACTIONS

19.1. INSTRUMENT RELATED

19.1.1. ICV not within $\pm$ 10% or $\pm$ 5% for 200.7
 a. Is the problem with the solution?
  i. Re-prepare or obtain new stock.

b. Is the problem with the calibration?
   i. Recalibrate through analysis of appropriate standards and recheck ICV.

19.1.2. ICB not $\pm$MDL or within $\pm$ 3X IDL or CRDL for CLP, **DOD no analytes detected >LOD**

a. Is the problem with the solution?
   i. Re-prepare
b. Is the problem with the calibration?
   i. Recalibrate with the blank solution or the low level standard. Restart analysis with the ICV.

19.1.3. Check standards not within $\pm$ 5%

a. Is the problem with the solution?
   i. Re-pour, re-prepare or obtain new stock.
b. Is the problem with the calibration?
   i. Recalibrate thru analysis of appropriate standards. Restart analysis with the ICV.

19.1.4. CLP only-CRI not within $\pm$ 20% (Internal QC, only required for CLP work).

a. Is the problem with the solution?
   i. Re-prepare or obtain new stock.
b. Is the problem with the calibration?
   i. Recalibrate thru analysis of appropriate standards. Restart analysis with the ICV.

19.1.5. IFA metals not present are not less than the detection limit for that metal, **for IFA DOD, absolute value of concentration for all non-spiked analytes <±LOD.**

a. Is the problem with the solution?
   i. Re-prepare or obtain new stock.
b. Is the problem with the calibration?
   i. Recalibrate thru analysis of appropriate standards. Restart analysis with the ICV.

19.1.6. IFB not within $\pm$ 20%

a. Is the problem with the solution?
   i. Re-prepare or obtain new stock.
b. Is the problem with the calibration?
   i. Recalibrate thru analysis of appropriate standards. Restart analysis with the ICV.

19.1.7. CCV not within $\pm$ 10%

a. Is the problem with the solution?
   i. Re-prepare or obtain new stock.
b. Is the problem with the calibration?
   i. If appropriate, continue the analysis. Discuss effect of the out of control situation with your supervisor. The samples will be reanalyzed or the data will be qualified.

19.1.8.. CCB not ±2xMDL or CRDL for CLP, DOD no analytes detected >±LOD.
  a. Is the problem with the solution?
    i. Re-prepare
  b. Is the problem with the calibration?
    i. Re-calibrate and reanalyze.

19.2. DIGESTION RELATED

19.2.1. Preparation blank (BLK) not within ± ½ RL and ± RL for common contaminants DOD or RL/CRDL for other or CLP
  a. Is the problem with the instrument?
    i. Evaluate with respect to instrumental bias or reanalyze when instrument is in control.
  b. Is the problem with the digestion?
    i. If associated samples are less than 10X the level of the preparation blank but above the RL, the sample must be re-digested or the data must be qualified on the final report.

19.2.2. BS not within control limits
  a. Is the problem with the instrument?
    i. Evaluate with respect to instrumental bias or reanalyze when instrument is in control.
  b. Is the problem with the digestion?
    i. If biased low, associated samples must be re-digested.
    ii. If biased high, the impact upon the data user must be evaluated. The samples will be re-digested or the data will be qualified on the final report.

19.3. SAMPLE MATRIX RELATED

19.3.1. Replicate analysis RPD not within ±20% (if both are >5X CRDL) or ± the CRDL (if either are <5X CRDL).
  a. The associated sample data must be qualified on the final report.

19.3.2. Spike analysis recovery not within ±20%.
  a. Is the analyte level in the sample greater than 4X the spiking level?
    i. If yes, the spike recovery is not evaluated.
    ii. If no, a post digestion spike must be analyzed and the associated sample data must be qualified on the final report.

19.3.3. When required, post digestion spike analysis recovery not within ±25% for SW6010B, DOD or ± 20% SW6010C.
  a. The associated sample data must be qualified on the final report.
  b. For USACE analysis by MSA is required.

19.3.4. Serial dilution analysis percent difference not within ±10%
  a. Is the analyte concentration a factor of 50 above the instrumental detection limit after dilution?

i. If no, the serial dilution data can not be evaluated.

iii. If yes, a chemical or physical interference effect should be suspected. The analyst and or section manager must note this situation on the final analytical report.

20. **Waste Management**

Laboratory SOP QS14 on Waste Handling discusses general guidelines for the appropriate handling of wastes and the laboratory program on waste management.

21. **References**

21.1. *Test Methods for Evaluating Solid Waste Physical/Chemical Methods, SW-846; Third Edition (Update III)*; Method 6010B and Method 6010C.

21.2. USEPA *Code of Federal Reqlations, 40, CH 1,PT 136;* Method 200.7; APX-B.

21.3. USEPA *Contract Laboratory Program (CLP) for Inorganics ILM04.1; ILM05.2*

21.4. DOD Quality Systems Manual for Environmental Laboratories Version 4.1. (Based on NELAC Voted Revision June 5, 2003. 4/22/09

22. **Tables, Diagrams, Flowcharts and Validation Data**

Table 1 contains all applicable parameters with the applicable RL/LOQ, LOD and Detection Limit.

Table 1A, contains a list of the wavelengths used for each analyte.

Table 2, for all technical methods, contains the QA/QC summary table.

Table 3, Technical Completeness / Accuracy Checklist

Table 4, Data Reviewers Checklist

Table 1 Water				
Analyte	MDL	LOD	MRL	Units
Aluminum	50.0	100	200	ug/L
Antimony	5.00	8.00	15.0	ug/L
Arsenic	3.00	6.00	10.0	ug/L
Barium	5.00	10.0	40.0	ug/L
Beryllium	1.00	2.00	5.00	ug/L
Boron	10.0	20.0	30.0	ug/L
Cadmium	1.00	2.00	5.00	ug/L
Calcium	1000	2000	5000	ug/L
Chromium	2.00	4.00	10.0	ug/L
Cobalt	5.00	10.0	12.5	ug/L
Copper	4.00	8.00	10.0	ug/L
Iron	30.0	60.0	100	ug/L
Lead	1.50	3.00	3.00	ug/L
Magnesium	1000	3000	5000	ug/L
Manganese	3.00	6.00	15.0	ug/L
Molybdenum	5.00	10.0	15.0	ug/L
Nickel	3.00	6.00	10.0	ug/L
Potassium	1000	3000	5000	ug/L
Selenium	3.00	5.00	6.00	ug/L
Silver	1.00	2.00	10.0	ug/L
Sodium	1000	3000	5000	ug/L
Thallium	3.00	4.00	8.00	ug/L
Tin	10.0	20.0	30.0	ug/L
Titanium	5.00	10.0	15.0	ug/L
Vanadium	5.00	10.0	12.5	ug/L
Zinc	5.00	10.0	20.0	ug/L

Table 1 TCLP				
Analyte	MDL	LOD	MRL	Units
Antimony	0.00500	0.00800	0.0150	mg/L
Arsenic	0.00300	0.00600	0.0100	mg/L
Barium	0.00500	0.0100	0.0400	mg/L
Cadmium	0.00100	0.00200	0.00500	mg/L
Chromium	0.00200	0.00400	0.0100	mg/L
Copper	0.00400	0.00800	0.0100	mg/L
Lead	0.00150	0.00300	0.00300	mg/L
Selenium	0.00300	0.00500	0.00600	mg/L
Silver	0.00100	0.00200	0.0100	mg/L

Table 1 Soil				
Analyte	MDL	LOD	MRL	Units
Aluminum	10.0	20.0	40.0	mg/Kg
Antimony	1.00	1.60	3.00	mg/Kg
Arsenic	0.600	1.20	2.00	mg/Kg
Barium	1.00	2.00	8.00	mg/Kg
Beryllium	0.200	0.400	1.00	mg/Kg
Boron	2.00	4.00	6.00	mg/Kg
Cadmium	0.200	0.400	1.00	mg/Kg
Calcium	200	400	1000	mg/Kg
Chromium	0.400	0.800	2.00	mg/Kg
Cobalt	1.00	2.00	2.50	mg/Kg
Copper	0.800	1.60	2.00	mg/Kg
Iron	6.00	12.0	20.0	mg/Kg
Lead	0.300	0.600	0.600	mg/Kg
Magnesium	200	600	1000	mg/Kg
Manganese	0.600	1.20	3.00	mg/Kg
Molybdenum	1.00	2.00	3.00	mg/Kg
Nickel	0.600	1.20	2.00	mg/Kg
Potassium	200	600	1000	mg/Kg
Selenium	0.600	1.00	1.20	mg/Kg
Silver	0.200	0.400	2.00	mg/Kg
Sodium	200	600	1000	mg/Kg
Thallium	0.600	0.800	1.60	mg/Kg
Tin	2.00	4.00	6.00	mg/Kg
Titanium	1.00	2.00	3.00	mg/Kg
Vanadium	1.00	2.00	2.50	mg/Kg
Zinc	1.00	2.00	4.00	mg/Kg

TABLE 1A

METAL	WAVELENGTH
Aluminum	396.1
Antimony	206.8
Arsenic	189.0
Barium	233.5
Beryllium	313.0
Boron	249.7
Cadmium	228.8
Calcium	317.9
Chromium	267.7
Cobalt	228.6
Copper	324.7
Iron	261.1
Lead	220.3
Magnesium	279.0
Manganese	257.6
Molybdenum	202.0
Nickel	231.6
Potassium	766.4
Selenium	196.0
Silver	328.0
Sodium	589.5
Strontium	421.5
Thallium	190.8
Tin	189.9
Titanium	334.9
Vanadium	292.4
Zinc	206.2

Table 2 - Method Quality Control Requirements Summary			
QC Check	**Minimum Frequency / Requirements**	**Acceptance Criteria**	**Corrective Action for Failures / Data Useability**
Interference Check	• once per calibration	• IFA less than LOD if not verified contamination of standard. IFB must be within ±20%.	• Check IEC corrections for metals in the IFA.
Calibration Curve	• Prior to analyzing any samples • A minimum of a blank and 3-points for linear fits client specific requirement or a blank and high standard. • Low standard at the RL level run against the curve within 20% initially and within 30% for subsequent analysis (6010C).	• Linear calibration Corr. of 0.998 • Must follow curve processing requirements from SOP QS08	• Re-evaluate curve mix and makeup • Re-run curve • Check instrument for maintenance needs • Re-prep the curve standards Samples cannot be analyzed until there is a passing calibration
ICB	At the beginning of every sequence	Must meet the <±LOD for DOD or < 2xMDL	Re-run
ICV	Alternate source standard to be analyzed after every calibration curve	• Must be in the range 90 to 110% for 6010B&C, or 95 to 115% for 200.7.	• Re-analyze an ICV from a different source • Re-prep and re-analyze the ICV • Re-calibrate and verify standard preps and sources
CCV	• At the beginning of every sequence • For every 10-client samples	• Must be in the range 90 to 110%	• Samples must be reanalyzed if possible, if not samples are flagged with a “Q”.
Closing CCV	• At the end of every sequence	• Must be in the range 90 to 110%	• Samples must be reanalyzed if possible, if not samples are flagged with a “Q”.
BLK	One per prep batch	• Must be less than ½ ±RL.	• Re-analysis to confirm the positive value • Ascertain if there are any samples within the batch that meet the MB criteria and provide the information for the decision makers • If results are between the LOD or RL/LOQ, then assess the data and notify the PM for further action • Re-prep of samples associated with the MB • NCR will be required for data reported • Final Report data flagging will be required

Table 2 - Method Quality Control Requirements Summary			
QC Check	**Minimum Frequency / Requirements**	**Acceptance Criteria**	**Corrective Action for Failures / Data Useability**
BS	One per prep batch	Must be in the range of 80 to 120% for 6010B, DOD; or 85 to 115% for 200.7.	• Rerun to confirm problem. • All samples associated with the LCS must be re-digested, reanalyzed if possible. • NCR will be required for data reported • If samples cannot be re-digested or re-analyzed Final Report data flagging will be required
MS	One per prep batch	Must be in the range of 80 to 120%	Final Report data flagging will be required
MSD	One per prep batch	Must be in the range of 80 to 120%	Final Report data flagging will be required
Sample Duplicate	One per prep batch	20%	Flag samples
Post Digestion Spike	One per batch	±25% for DOD/6010B, ±20% 6010C	If possible MSA required, Flag samples
DOC Study	• Initially per analyst prior to reporting data • Annually • Follow specific guidelines from section 16 for the preparation and analysis of DOC samples	• Must meet the criteria of the BS for average accuracy	• Re-prep and / or • Re-analysis
MDL Study	Once per year		
LOD Verification	Every quarter		
LOQ Verification	Every quarter		
Linear Dynamic Range Study (LDR)	Every six months		

Table 3, Technical Completeness / Accuracy Checklist

1. Were all the QC check elements analyzed – refer to Table 2 of the SOP
2. Were the QC criteria met
3. In cases of failures, was there an NCR written
4. Were dilution factors applied correctly
5. Was the data uploaded into LIMS via direct upload – if yes, then was a cross check subset of the uploaded values performed
6. Was the red marked data in LIMS checked for accuracy and the corresponding hard copy data documented appropriately
7. Were proper data qualifiers applied to the data in LIMS
8. Was the hard copy package checked for completeness to include all data for the sequence such that the data reviewer could reconstruct sample analyses and validate / approve the data

Table 4, Data Reviewers Checklist (Prior to approving data)

1. Does the hard copy raw data (or electronic raw data) package look complete and include all data points
2. Were QA objectives met and for failures were the appropriate actions taken
3. For direct uploads to LIMS, did a subset cross check match the raw data
4. Did all the manual entries into LIMS match the raw data
5. Were there appropriate signatures and documentation on the raw data
6. Were appropriate LIMS flags used
7. Were manual calculations verified

ANALYST DATA REVIEW CHECKLIST Sample Number(s):
Batch Number(s):
Method: 6010B or 6010C (ICP)

QA/QC Item	Yes	No	NA	Second Level Review
1. Were samples analyzed within USACE holding times?				
2. Was initial calibration curve QC criteria met?				
3. Was all continuing calibration criteria in control?				
4. Did any sample exceed the highest calibration standard? (If yes, were appropriate dilutions made to generate samples concentration within calibration range?)				
5. Did BS or blank spike meet control limits?				
6. Did MS/MSD meet control limits?				
7. Was the preparation (Method) Blank (BLK) below the project required detection limits?				
8. Did you return samples back to cold storage immediately after use?				
9. Was hot plate temperature monitored/documented and did you apply the thermometer correction factor?				
10. Sample preparation information is correct and complete.				
11. Analytical results are correct and complete.				
12. The appropriate SOP's have been used and followed.				
14. "Raw data" including all manual integration's have been correctly interpreted.				
15. "Special" sample preparation and analytical requirements have been met.				
16. Documentation complete (e.g., all anomalies in the analytical sequence have been documented, corrective action forms are complete.				

Comments on any "No" response:

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Analyst: ______________________ Date: ______________________

SYNTHETIC PRECIPITATION LEACHING PROCEDURE

METHOD 1312

SOP NUMBER: SOP-147

REVISION NUMBER: 0

APPROVED BY:

Betty DeVille
SECTION MANAGER

Randy D. Ward
TECHNICAL DIRECTOR

EFFECTIVE DATE: 10/22/02

DATE OF LAST REVIEW: 05/26/09

METHOD 1312

SYNTHETIC PRECIPITATION LEACHING PROCEDURE

1.0 SCOPE AND APPLICATION

1.1 The SPLP is designed to determine the mobility of both organic and inorganic analytes present in liquids, soils, and wastes.

2.0 SUMMARY OF METHOD

2.1 For liquid samples, (i.e., those containing less than 0.5% dry solid material), the sample, after filtration through a 0.6 to 0.8 um glass fiber filter, is defined as the 1312 extract.

2.2 For samples containing greater than 0.5% solids, the liquid phase, if any, is separated from the solid phase and stored for later analysis; the particle size of the solid phase is reduced, if necessary. The solid phase is extracted with an amount of extraction fluid equal to 20 times the weight of the solid phase. The extraction fluid employed is a function of the region of the country where the sample site is located if the sample is a soil. If the sample is a waste or wastewater, the extraction fluid employed is a pH 4.2 solution. A special extractor vessel is used when testing for volatile analytes. Following extraction, the liquid extract is separated from the solid phase by filtration through a 0.6 to 0.8 um glass fiber filter.

2.3 If compatible (i.e., multiple phases will not form on combination), the initial liquid phase of the waste is added to the liquid extract, and these are analyzed together. If incompatible, the liquids are analyzed separately and the results are mathematically combined to yield a volume-weighted average concentration.

3.0 INTERFERENCES

3.1 Potential interferences that may be encountered during analysis are discussed in the individual analytical methods and related SOPs. (See section 2.2)

4.0 APPARATUS AND MATERIALS

4.1 Agitation apparatus: The agitation apparatus must be capable of rotating the extraction vessel in an end-over-end fashion at 30 $\pm$ 2 rpm. This rate is measured and recorded with each extraction to ensure accuracy of the equipment.

4.2 Extraction Vessels

4.2.1 Zero-Headspace Extraction Vessel (ZHE). This device is for use only when the sample is being tested for the mobility of volatile analytes. The ZHE allows for liquid/solid separation within the device, and effectively precludes headspace. This type of vessel allows for initial liquid/solid separation, extraction, and final extract filtration without opening the vessel. These vessels shall have an internal volume of 500-600 mL, and be equipped to accommodate a 90-110 mm filter. The devices contain VITON®[1] O-rings which should be replaced frequently.

For the ZHE to be acceptable for use, the piston within the ZHE should be able to be moved with approximately 15 psig or less. If it takes more pressure to move the piston, the O-rings in the device should be replaced. If this does not solve the problem, the ZHE is unacceptable for 1312 analyses and the manufacturer should be contacted.

The ZHE should be checked for leaks after every extraction. If the device contains a built-in pressure gauge, pressurize the device to 50 psig, allow it to stand unattended for 1 hour, and recheck the pressure. If the device does not have a built-in pressure gauge, pressurize the device to 50 psi, submerge it in water, and check for the presence of air bubbles escaping from any of the fittings. If pressure is lost, check all fittings and inspect and replace O-rings, if necessary. Retest the device. If leakage problems cannot be solved, the manufacturer should be contacted.

Some ZHEs use gas pressure to actuate the ZHE piston, while others use mechanical pressure. Whereas the volatiles procedure refers to pounds per square inch (psi), for the mechanically actuated piston, the pressure applied is measured in torque-inch-pounds.

4.2.2 Bottle Extraction Vessel. When the sample is being evaluated using the nonvolatile extraction, a jar with sufficient capacity to hold the sample and the extraction fluid is needed. Headspace is allowed in this vessel.

The extraction bottles may be constructed from various materials, depending on the analytes to be analyzed and the nature of the waste. It is recommended that borosilicate glass bottles be used instead of other types of glass, especially when inorganics are of concern. Plastic bottles, other than polytetrafluoroethylene, shall not be used if organics are to be investigated.

4.3 Filtration Devices: It is recommended that all filtrations be performed in a hood or well ventilated area.

4.3.1 Zero-Headspace Extractor Vessel (ZHE): When the sample is evaluated for volatiles, the zero-headspace extraction vessel is used for filtration. The device shall be capable of supporting and keeping in place the glass fiber filter and be able to withstand the pressure needed to accomplish separation (50 psig).

NOTE: When it is suspected that the glass fiber filter has been ruptured, an in-line glass fiber filter may be used to filter the material within the ZHE.

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[1] VITON® is a trademark of Du Pont.

4.3.2 Filter Holder: When the sample is evaluated for other than volatile analytes, a filter holder capable of supporting a glass fiber filter and able to withstand the pressure needed to accomplish separation may be used. Suitable filter holders range from simple vacuum units to relatively complex systems capable of exerting pressures of up to 50 psi or more. Vacuum filtration can only be used for wastes with low solids content (<10%) and for highly granular, liquid-containing wastes. All other types of wastes should be filtered using positive pressure filtration.

4.3.3 Materials of Construction: Extraction vessels and filtration devices shall be made of inert materials which will not leach or absorb sample components of interest. Glass, polytetrafluoroethylene (PTFE), or type 316 stainless steel equipment may be used when evaluating the mobility of both organic and inorganic components.

4.4 Filters: Filters shall be made of borosilicate glass fiber, shall contain no binder materials, and shall have an effective pore size of 0.6 to 0.8 µm (Whatman GF/F), or equivalent. **Pre-filters must not be used. When evaluating the mobility of metals**, filters shall be acid-washed prior to use by rinsing with 1N nitric acid followed by three consecutive rinses with deionized distilled water (a minimum of 1 L per rinse is recommended). Glass fiber filters are fragile and should be handled with care.

4.5 pH Meters: The meter should be accurate to ± 0.05 units at 25°C.

4.6 ZHE Extract Collection Devices: VOA vials are used to collect the initial liquid phase and the final extract of the waste when using the ZHE device.

4.7 ZHE Extraction Fluid Transfer Devices: Any device capable of transferring the extraction fluid into the ZHE without changing the nature of the extraction fluid is acceptable (e.g., a positive displacement or peristaltic pump, a gas tight syringe, pressure filtration unit (see Section 4.3.2), or other ZHE device).

4.8 Laboratory Balance: Accurate to within ± 0.01 grams may be used (all weight measurements are to be within ± 0.1 grams).

4.9 Beaker or Erlenmeyer flask, glass, various sizes.

4.10 Watchglass

4.11 Magnetic stirrer.

5.0 REAGENTS

5.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

5.2 Reagent Water. Reagent water is defined as water in which an interferant is not observed at or above the method's detection limit of the analyte(s) of interest. For nonvolatile extractions, ASTM Type II water or equivalent meets the definition of reagent water. For volatile extractions, it is recommended that reagent water be generated by any of the following methods. Reagent water should be monitored periodically for impurities.

5.2.1 A water purification system may also be used to generate reagent water for volatile extractions.

5.2.2 Reagent water for volatile extractions may also be prepared by boiling water for 15 minutes. Subsequently, while maintaining the water temperature at 90 $\pm$ 5 degrees C, bubble a contaminant-free inert gas (e.g. nitrogen) through the water for 1 hour. While still hot, transfer the water to a narrow mouth screw-cap bottle under zero-headspace and seal with a Teflon-lined septum and cap.

5.3 Sulfuric acid/nitric acid (60/40 weight percent mixture) H_2SO_4/HNO_3. Cautiously mix 60 g of concentrated sulfuric acid with 40 g of concentrated nitric acid. If preferred, a more dilute H_2SO_4/ HNO_3 acid mixture may be prepared and used in steps 5.4.1 and 5.4.2 making it easier to adjust the pH of the extraction fluids.

5.4 **Extraction fluids.**

5.4.1 Extraction fluid #1: This fluid is made by adding the 60/40 weight percent mixture of sulfuric and nitric acids (or a suitable dilution) to reagent water until the pH is 4.20 ± 0.05. The fluid is used to determine the leachability of soil from a site that is east of the Mississippi River, and the leachability of wastes and wastewaters. Determine the pH and record in appropriate reagent log (RL) notebook.

NOTE: Solutions are unbuffered and exact pH may not be attained.

5.4.2 Extraction fluid #2: This fluid is made by adding the 60/40 weight percent mixture of sulfuric and nitric acids (or a suitable dilution) to reagent water until the pH is 5.00 ±0.05. The fluid is used to determine the leachability of soil from a site that is west of the Mississippi River. Record in appropriate reagent log (RL) notebook.

5.4.2 Extraction fluid #3: This fluid is reagent water and is used to determine cyanide and volatiles leachability.

NOTE: These extraction fluids should be monitored frequently for impurities. The pH should be checked prior to use to ensure that these fluids are made up accurately. If impurities are found or the pH is not within the above specifications, the fluid shall be discarded and fresh extraction fluid prepared.

5.5 Analytical standards shall be prepared according to the appropriate analytical method.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 All samples shall be collected using an appropriate sampling plan.

6.2 There may be requirements on the minimal size of the field sample, depending upon the physical state or states of the waste and the analytes of concern. An aliquot is needed for the preliminary evaluations of the percent solids and the particle size. An aliquot may be needed to conduct the nonvolatile analyte extraction procedure. If volatile organics are of concern, another analyte may be needed. Quality control measures may require additional aliquots. Further it is always wise to collect more sample just in case something goes wrong with the initial attempt to conduct the test.

6.3 Preservatives shall not be added to samples before extraction.

6.4 Samples may be refrigerated unless refrigeration results in irreversible physical change tot the waste. If precipitation occurs the entire sample (including precipitate) should be extracted.

6.5 When the sample is to be evaluated for volatile analytes, care shall be taken to minimize the loss of volatiles. Samples shall be collected and stored in a manner intended to prevent the loss of volatile analytes (e.g., samples should be collected in Teflon -lined septum capped vials and stored at 4°C. Samples should be opened only immediately prior to extraction.

6.4 For volatiles, the samples shall be collected and stored in a manner intended to prevent the loss of volatile analytes (e.g., samples should be collected in Teflon-lined septum capped vials and stored at 4°C. Samples should be opened only immediately prior to extraction).

6.5 1312 extracts should be prepared for analysis and analyzed as soon as possible following extraction. **Extracts or portions of extracts for metallic analyte determinations must be acidified with nitric acid to a pH < 2, unless precipitation occurs (see Section 7.2.14 if precipitation occurs).** Extracts should be preserved for other analytes according to the guidance given in the individual analysis methods. Extracts or portions of extracts for organic analyte determinations shall not be allowed to come into contact with the atmosphere (i.e., no headspace) to prevent losses. See step 8.0 (Quality Control) for acceptable sample and extract holding times.

7.0 PROCEDURE

Care should be used in handling all samples. Safety glasses must be worn in the lab at all times. The use of blue nitrile gloves and lab coats is highly recommended.

Research into expected sample content and concentration should be done in order to be prepared for additional safety considerations. Generally, any samples which need special consideration have applicable notes on the sample logs.

MSDS are available for all reagents and standards, which have been purchased. These are located in the office next to the technical director.

7.1 Preliminary Evaluations

Perform preliminary 1312 evaluations on a minimum 100 gram aliquot of sample. This aliquot may not actually undergo 1312 extraction. These preliminary evaluations include: (1) determination of the percent solids (Step 7.1.1); (2) determination of whether the waste contains insignificant solids and is, therefore, its own extract after filtration (Step 7.1.2); (3) determination of whether the solid portion of the waste requires particle size reduction. (Step 7.1.3).

7.1.1 Preliminary determination of percent solids: Percent solids is defined as that fraction of a waste sample (as a percentage of the total sample) from which no liquid may be forced out by an applied pressure as described below.

7.1.1.1 If the sample will obviously yield no free liquid when subjected to pressure filtration, weigh out a representative subsample (100 g minimum) and proceed to Step 7.1.3.

7.1.1.2 If the sample is liquid or multiphasic, liquid/solid separation to make a preliminary determination of percent solids is required. This involves the filtration device discussed in Section 4.3.2 and is outlined in Sections 7.1.1.3 through 7.1.1.9.

7.1.1.3 Pre-weigh the filter and the container that will receive the filtrate.

7.1.1.4 Assemble the filter holder and filter following the manufacturer's instructions. Place the weighed filter in the buchner funnel.

7.1.1.5 Mix the sample and weigh out a representative subsample of (100gram minimum) and record exact weight.

7.1.1.6 Allow sludges to stand to permit the solid phase to settle. Samples that settle slowly may be centrifuged prior to filtration. Centrifugation is to be used only as an aid to filtration. If used, the liquid should be decanted and filtered followed by filtration of the solid portion of the waste through the same filtration system.

7.1.1.7 Quantitatively transfer the sample to the filter holder (liquid and solid phases). Spread the waste sample evenly over the surface of the filter. If filtration of the waste at 4°C reduces the amount of expressed liquid over what would be expressed at room temperature, then allow the sample to warm up to room temperature in the device before filtering.

Gradually apply vacuum or gentle pressure of 1-10 psi, until air or pressurizing gas moves through the filter. If this point is not reached under 10 psi, and if no additional liquid has passed through the filter in any 2 minute interval, slowly increase the pressure in 10 psi increments to a maximum of 50 psi. After each incremental increase of 10 psi, if the pressurizing gas has not moved through the filter, and if no additional liquid has passed through the filter in any 2 minute interval, proceed to the next 10 psi increment. When the pressurizing gas begins to move through the filter, or when liquid flow has ceased at 50 psi (i.e., filtration does not result in any additional filtrate within any 2 minute period), stop the filtration.

NOTE: If sample material (>1% of original sample weight) has obviously adhered to the container used to transfer the sample to the filtration apparatus, determine the weight of this residue and subtract it from the sample weight determined in Section 7.1.1.5 to determine the weight of the waste sample that will be filtered.

7.1.1.8 The material in the filter holder is defined as the solid phase of the waste, and the filtrate is defined as the liquid phase.

NOTE: Some samples, such as oily wastes and some paint wastes, will obviously contain some material that appears to be a liquid but even after applying vacuum or pressure filtration, as outlined in Step 7.1.1.7, this material may not filter. If this is the case, the material within the filtration device is defined as a solid. Do not replace the original filter with a fresh filter under any circumstances. Use only one filter.

7.1.1.9 Determine the weight of the liquid phase by subtracting the weight of the filtrate container (see section 7.1.1.3) from the total weight of the filtrate-filled container. Determine the weight of the solid phase of the sample by subtracting the weight of the liquid phase from the weight of the total sample, as determined in Section 7.1.1.5 or 7.1.1.7.

Record the weight of the liquid and solid phases. Calculate the percent solids as follows:

$$\text{Percent solids} = \frac{\text{Weight of solid}}{\text{Total weight of waste}} \times 100$$

7.1.2 If the percent solids determined in Step 7.1.1.9 is equal to or greater than 0.5%, then proceed either to Step 7.1.3 to determine whether the solid material requires particle size reduction or to Step 7.1.2.1 if it is noticed that a small amount of the filtrate is entrained in wetting of the filter. If the percent solids is less than 0.5%, then proceed to Step 7.2.9 if the nonvolatile 1312 analysis is to be performed and to Section 7.3 with a fresh portion of the waste if the volatile 1312 is to be performed.

7.1.2.1 Remove the solid material and filter from the filtration apparatus.

7.1.2.2 Dry the filter and solid material at 100 $\pm$ 20°C until 2 successive weighings yield the same value within ±1%. Record the final weight.

Caution: should be taken to ensure that the subject solid will not flash upon heating. It is recommended that the drying oven be vented to a hood or other appropriate device.

7.1.2.3 Calculate the percent dry solids as follows:

$$\text{Percent dry solids} = \frac{\text{(Wt. of dry waste + filter) - tared wt. of filter}}{\text{Initial wt. of waste}} \times 100$$

7.1.2.4 If the percent dry solids is less than 0.5%, then proceed to Step 7.2.9 if the nonvolatile 1312 analysis is to be performed, and to Step 7.3 if the volatile 1312 is to be performed. If the percent dry solids is greater than or equal to 0.5%, and if the nonvolatile 1312 analysis is to be performed, return to the beginning of this Step (7.1) and, with a fresh portion of sample, determine whether particle size reduction is necessary (Step 7.1.3).

7.1.3 Determination of whether the sample requires particle-size reduction: Using the solid portion of the sample, evaluate the solid for particle size. Particle size reduction is required, unless the solid passes through a 9.5 mm (0.375 inch) standard sieve). If the surface area is smaller or the particle size larger than described above, prepare the solid portion of the waste for extraction by crushing, cutting, or grinding the waste to a surface area or particle size as described above. If the solids are prepared for organic volatiles extraction, special precautions must be taken. (see Step 7.3.6).

NOTE: Surface area criteria are meant for filamentous (e.g., paper, cloth, and similar) waste materials. Actual measurement of surface area is not required, nor is it recommended. For materials that do not obviously meet the criteria, sample-specific methods would need to be developed and employed to measure the surface area. Such methodology is currently not available.

7.1.4 Determination of appropriate extraction fluid:

7.1.4.1 For soils, if the sample is from a site that is east of the Mississippi River, extraction fluid #1 should be used. If the sample is from a site that is west of the Mississippi River, extraction fluid #2 should be used.

7.1.4.2 For wastes and wastewater, extraction fluid #1 should be used.

7.1.4.3 For cyanide containing wastes and/or soils, extraction fluid #3 (reagent water) must be used because leaching of cyanide containing samples under acidic conditions may result in the formation of hydrogen cyanide gas.

7.1.5 If the aliquot of the waste used for the preliminary evaluation was determined to be 100% solid, then it can be used for the Step 7.2 extraction (assuming at least 100 grams remain), and the Step 7.3 extraction (assuming at least 25 grams remain). If the aliquot was subjected to the procedure in Step 7.1.1.7, then another aliquot shall be used for the volatile extraction procedure in step 7.3. The aliquot of the waste subjected to the procedure in step 7.1.1.7. might be appropriate for use for the Step 7.2 extraction if an adequate amount of solid (as determined by Step 7.1.1.9) was obtained. The amount of solid necessary is dependent upon whether a sufficient amount of extract will be produced to support the analyses. If an adequate amount of solid remains, proceed to Step 7.2.10 of the nonvolatile 1312 extraction.

7.2 Procedure When Volatiles Are Not Involved

A minimum sample size of 100 grams (solid and liquid phases) is recommended. If less than the specified amount is used, proportion extraction fluid accordingly and notify manager. In some cases, a larger sample size may be appropriate, depending on the solids content of the waste sample, whether the initial liquid phase of the waste will be miscible with the aqueous extract of the solid, and whether inorganics, semivolatile organics, pesticides, and herbicides are all analytes of concern. Enough solids should be generated for extraction such that the volume of 1312 extract will be sufficient to support all of the analyses required. If the amount of extract generated by a single 1312 extraction will not be sufficient to perform all of the analyses, more than one extraction may be performed and the extracts from each combined and aliquoted for analysis.

7.2.1 If the sample will obviously yield no liquid when subjected to pressure filtration (i.e., is 100% solid, see step 7.1.1), weigh out a subsample of the sample (100 gram minimum) and proceed to step 7.2.9.

7.2.2 If the sample is liquid or multiphasic, liquid/solid separation is required. This involves the filtration device described in step 4.3.2 and is outlined in steps 7.2.3 to 7.2.8.

7.2.3 Pre-weigh the container that will receive the filtrate.

7.2.4 Assemble the filter holder and filter following the manufacturer's instructions. Place the filter on the support screen and secure. Acid wash the filter if evaluating the mobility of metals (see step 4.4).

NOTE: Acid washed filters may be used for all nonvolatile extractions even when metals are not of concern.

7.2.5 Weigh out a subsample of the sample (100 gram minimum) and record the weight. If the waste contains <0.5% dry solids (step 7.1.2), the liquid portion of the waste, after filtration, is defined as the 1312 extract. Therefore, enough of the sample should be filtered so that the amount of filtered liquid will support all of the analyses required of the 1312 extract. For wastes containing >0.5% dry solids (Steps 7.1.1 or 7.1.2), use the percent solids information obtained in Step 7.1.1 to determine the optimum sample size (100 gram minimum) for filtration. Enough solids should be generated by filtration to support the analyses to be performed on the 1312 extract.

7.2.6 Allow slurries to stand to permit the solid phase to settle. Samples that settle slowly may be centrifuged prior to filtration. Use centrifugation only as an aid to filtration. If the sample is centrifuged, the liquid should be decanted and filtered followed by filtration of the solid portion of the waste through the same filtration system.

7.2.7 Quantitatively transfer the sample (liquid and solid phases) to the filter holder (see step 4.3.2). Spread the waste sample evenly over the surface of the filter. If filtration of the waste at 4 °C reduces the amount of expressed liquid over what would be expressed at room temperature, then allow the sample to warm up to room temperature in the device before filtering.

Gradually apply vacuum or gentle pressure of 1-10 psig, until air or pressurizing gas moves through the filter. If this point if not reached under 10 psig, and if no additional liquid has passed through the filter in any 20minute interval, slowly increase the pressure in 10-psig increments to maximum of 50 psig. After each incremental increase of 10 psig, if the pressurizing gas has not moved through the filter, and if no additional liquid has passed through the filter in any 2-minute interval, proceed to the next 10-psig increment. When the pressurizing gas begins to move through the filter, or when the liquid flow has ceased at 50 psig (i.e., filtration does not result in any additional filtrate within a 2-minute period) a stop the filtration.

<u>NOTE</u>: If waste material (>1 % of the original sample weight) has obviously adhered to the container used to transfer the sample to the filtration apparatus, determine the weight of this residue and subtract it from the sample weight determined in step 7.2.5, to determine the weight of the waste sample that will be filtered.

<u>NOTE</u>: Instantaneous application of high pressure can degrade the glass fiber filter and may cause premature plugging.

7.2.8 The material in the filter holder is defined as the solid phase of the sample, and the filtrate is defined as the liquid phase. Weigh the filtrate. The liquid phase may now be either analyzed (see Step 7.2.12) or stored at 4°C until time of analysis.

NOTE: Some wastes, such as oily wastes and some paint wastes, will obviously contain some material which appears to be a liquid. Even after applying vacuum or pressure filtration, as outlined in Step 7.2.7, this material may not filter. If this is the case, the material within the filtration device is defined as a solid, and is carried through the extraction as a solid. Do not replace the original filter with a fresh filter under any circumstances. Use only one filter.

7.2.9 If the sample contains <0.5% dry solids (see Step 7.1.2), proceed to Step 7.2.13. If the sample contains >0.5% dry solids (see Step 7.1.1 or 7.1.2), and if particle-size reduction of the solid was needed in step 7.1.3, proceed to Step 7.2.10. If the sample as received passes a 9.5 mm sieve, quantitatively transfer the solid material into the extractor bottle along with the filter used to separate the initial liquid from the solid phase, and proceed to Step 7.2.11.

7.2.10 Prepare the solid portion of the sample for extraction by crushing, cutting, or grinding the waste to a surface area or particle-size as described in Step 7.1.3. When the surface area or particle-size has been appropriately altered, quantitatively transfer the solid material into an extractor bottle. Include the filter used to separate the initial liquid from the solid phase.

NOTE: Sieving of the waste is not normally required. Surface area requirements are meant for filamentous (e.g. paper, cloth) and similar waste materials. Actual measurement of surface area is not recommended. If sieving is necessary, a Teflon-coated sieve should be used to avoid contamination of the sample.

7.2.11 Determine the amount of extraction fluid to add to the extractor vessel as follows:

$$\text{Weight of extraction fluid} = \frac{20 \text{ x percent wet solids x weight of waste fltered}}{100}$$

Slowly add this amount of appropriate extraction fluid (see Step 7.1.4) to the extractor vessel. Close the extractor bottle tightly (it is recommended that Teflon tape be used to ensure a tight seal), secure in rotary extractor device, and rotate at 30 ±2 rpm for 18 ± 2 hours. Ambient temperature (i.e., temperature of room in which extraction takes place) shall be maintained at 23 ± 2°C during the extraction period.

NOTE: As agitation continues, pressure may build up within the extractor bottle for some types of sample (e.g. limed or calcium carbonate-containing sample may evolve gases such as carbon dioxide). To relieve excess pressure, the extractor bottle may be periodically opened (e.g., after 15 minutes, 30 minutes, and 1 hour) and vented into a hood.

7.2.12 Following the 18 (±2) hour extraction, separate the material in the extractor vessel into its component liquid and solid phases by filtering through a new glass fiber filter, as outlined in Step 7.2.7. For final filtration of the 1312 extract, the glass fiber filter may be changed, if necessary, to facilitate filtration. Filter(s) shall be acid-washed (see Step 4.4) if evaluating the mobility of metals.

7.2.13 Prepare the 1312 extract as follows:

7.2.13.1 If the sample contained no initial liquid phase, the filtered liquid material obtained form Step 7.2.12 is defined as the 1312 extract. Proceed to Step 7.2.14.

7.2.13.2 If compatible (e.g., multiple phases will not result on combination), combine the filtered liquid resulting from Step 7.2.12 with the initial liquid phase of the sample obtained in Step 7.2.7. This combined liquid is defined as the 1312 extract. Proceed to Step 7.2.14.

7.2.13.3 If the initial liquid phase of the waste, as obtained from Step 7.2.7, is not or may not be compatible with the filtered liquid resulting from Step 7.2.12, do not combine these liquids. Analyze these liquids, collectively defined as the 1312 extract, and combine the results mathematically, as described in Step 7.2.14.

7.2.14 Following collection of the 1312 extract, the pH of the extract must be recorded in the TCLP/SPLP logbook. Immediately aliquot and preserve the extract for analysis. Metals aliquots must be acidified with nitric acid to pH <2. If precipitation is observed upon addition of nitric acid to a small aliquot of the extract, then the remaining portion of the extract for metals analyses shall not be acidified and the extract shall be analyzed as soon as possible. All other aliquots must be stored under refrigeration (4°C) until analyzed. The 1312 extract shall be prepared and analyzed according to appropriate analytical methods. 1312 extracts to be analyzed for metals shall be acid digested by SW846 method 3010A except in those instances where digestion causes loss of metallic analytes. If an analysis of the undigested extract shows that the concentration of any regulated metallic analyte exceeds the regulatory level, then the waste is hazardous and digestion of the extract is not necessary. However, data on undigested extracts alone cannot be used to demonstrate that the waste is not hazardous. If the individual phases are to be analyzed separately, determine the volume of the individual phases (to ± 0.5%), conduct the appropriate analyses, and combine the results mathematically by using a simple volume-weighted average:

$$\text{Final Analyte Concentration} = \frac{(V_1)(C_1) + (V_2)(C_2)}{V_1 + V_2}$$

where:

V_1 = The volume of the first phase (L).
C_1 = The concentration of the analyte of concern in the first phase (mg/L).
V_2 = The volume of the second phase (L).
C_2 = The concentration of the analyte of concern in the second phase (mg/L).

7.2.15 Compare the analyte concentrations in the 1312 extract with the levels identified in the appropriate regulations. Refer to Section 8.0 for quality assurance requirements.

7.3 Procedure When Volatiles Are Involved

Use the ZHE device to obtain 1312 extract for analysis of volatile compounds only. Extract resulting from the use of the ZHE shall not be used to evaluate the mobility of non-volatile analytes (e.g., metals, pesticides, etc.).

The ZHE device has approximately a 500 mL internal capacity. The ZHE can thus accommodate a maximum of 25 grams of solid (defined as that fraction of sample from which no additional liquid may be forced out by an applied pressure of 50 psig), due to the need to add an amount of extraction fluid equal to 20 times the weight of the solid phase.

Charge the ZHE with sample only once and do not open the device until the final extract (of the solid) has been collected. Repeated filling of the ZHE to obtain 25 grams of solid is not permitted.

Do not allow the sample, the initial liquid phase, or the extract to be exposed to the atmosphere for any more time then is absolutely necessary. Any manipulation of these materials should be done when cold (4°C) to minimize loss of volatiles.

All glassware and equipment must be thoroughly cleaned before use. A hot soapy water wash and rinse with tap water, then repeat following with a DI water rinse. Rinse with methanol and a final rinse with DI water.

7.3.1 Preweigh the (evacuated) filtrate collection container (see Step 4.6)
7.2.2 If the sample is liquid or multiphasic, liquid/solid separation is required. This involves the filtration device described in Section 4.3.2 and is outlined in Sections 7.2.3 to 7.2.8.

7.2.3 Pre-weigh the container that will receive the filtrate.

7.2.4 Assemble the filter holder and filter following the manufacturer's instructions. Place the filter on the support screen and secure. Acid wash the filter if evaluating the mobility of metals (see Section 4.4).

7.2.5 Weigh out a subsample of the waste (100 gram minimum) and record the weight. If the waste contains <0.5% dry solids, the liquid portion, after filtration, is defined as the TCLP extract. Therefore, enough of the sample should be filtered so that the amount of filtered liquid will support all of the analyses required. For wastes containing >0.5% dry solids use the percent solids information to determine the optimum sample size (100 gram minimum) for filtration.

7.2.6 Allow sludges to stand to permit the solid phase to settle. Wastes that settle slowly may be centrifuged prior to filtration. Use centrifugation only as an aid to filtration. If the waste is centrifuged, the liquid should be decanted and filtered followed by filtration of the solid portion of the waste through the same filtration system.

7.2.7 Refer to Section 7.1.1.7.

7.2.8 Refer to Section 7.1.1.8.

7.2.9 If the waste contains <0.5% dry solids, proceed to Section 7.2.13. If the waste contains >0.5% dry solids, and if particle size reduction of the solid was needed in Section 7.1.3, proceed to Section 7.2.10. If the waste as received passes a 9.5 mm sieve, quantitatively transfer the solid material into the extractor bottle along with the filter used to separate the initial liquid from the solid phase, and proceed to Section 7.2.11.

7.2.10 Prepare the solid portion of the waste for extraction by crushing, cutting, or grinding the waste to a surface area or particle size as described in Section 7.1.3. When the surface area or particle size has been appropriately altered, quantitatively transfer the solid material into an extractor bottle. Include the filter used to separate the initial liquid from the solid phase.

NOTE: Sieving of the waste is not normally required. Surface area requirements are meant for filamentous (e.g., paper, cloth) and similar waste materials. Actual measurement of surface area is not recommended. If sieving is necessary, a Teflon coated sieve should be used to avoid contamination of the sample.

7.2.11 Determine the amount of extraction fluid to add to the extractor vessel as follows:

$$\text{Weight of extraction fluid} = \frac{20 \text{ x percent wet solids x weight of waste fltered}}{100}$$

Slowly add this amount of appropriate extraction fluid to the extractor vessel. Close the extractor bottle tightly (it is recommended that Teflon tape be used to ensure a tight seal), secure in rotary agitation device, and rotate at 30 $\pm$ 2 rpm for 18 $\pm$ 2 hours. Ambient temperature shall be maintained at 23 $\pm$ 2 C during the extraction period. Record both tumble rate and room temperature.

NOTE: As rotation continues, pressure may build up within the extractor bottle for some types of wastes. To relieve excess pressure, the extractor bottle should be periodically opened (e.g., after 15 minutes, and 30 minutes) and vented into a hood.

7.2.12 Following the 18 ± 2 hour extraction, separate the material in the extractor vessel into its component liquid and solid phases by filtering through a new glass fiber filter, as outlined in Section 7.2.7. For final filtration of the TCLP extract, the glass fiber filter may be changed, if necessary, to facilitate filtration. Filter(s) shall be acid-washed.

7.2.13 Prepare the TCLP extract as follows:

7.2.13.1 If the sample contained no initial liquid phase, the filtered liquid material obtained is defined as the TCLP extract. Proceed to Section 7.2.14.

7.2.13.2 If compatible (e.g., multiple phases will not result on combination), combine the filtered liquid resulting from Section 7.2.12 with the initial liquid phase of the waste obtained in Section 7.2.7. This combined liquid is defined as the TCLP extract. Proceed to Section 7.2.14.

7.2.13.3 If the initial liquid phase of the waste, as obtained from Section 7.2.7, is not or may not be compatible with the filtered liquid resulting from Section 7.2.12, do not combine these liquids. Analyze these liquids, collectively defined as the TCLP extract, and combine the results mathematically, as described in Section 7.2.14.

7.2.14 Following collection of the TCLP extract, the pH of the extract must be recorded. Immediately split aliquots, give to appropriate analyst for spiking and preservation of the extract for analysis. Metal aliquots must be acidified with nitric acid to pH <2. (If precipitation is observed upon addition of nitric acid to a small aliquot of the extract, then the remaining portion of the extract for metals analyses shall not be acidified and the extract shall be analyzed as soon as possible.) All other aliquots must be stored under refrigeration (4°C) until analyzed. TCLP extracts to be analyzed for metals shall be acid digested except in those instances where digestion causes loss of metallic analytes. Refer also to sections 2.2 and 6.5.

If the individual phases are to be analyzed separately, determine the volume of the individual phases (to ± 0.5%), conduct the appropriate analyses, and combine the results mathematically by using a simple volume-weighted average:

$$\text{Final Analyte Concentration} = \frac{(V_1)(C_1) + (V_2)(C_2)}{V_1 + V_2}$$

where:

V_1 = The volume of the first phase (L).
C_1 = The concentration of the analyte of concern in the first phase (mg/L).

V_2 = The volume of the second phase (L).
C_2 = On next page
C_2 = The concentration of the analyte of concern in the second phase (mg/L).

7.3 Volatiles Extraction Procedure

Use ZHE device to obtain TCLP extract for analysis of volatile compounds only.

The ZHE device has approximately a 500 mL internal capacity. The ZHE can thus accommodate a maximum of 25 grams of solid due to the need to add an amount of extraction fluid equal to 20 times the weight of the solid phase.

Charge the ZHE with sample only once and do not open the device until the final extract has been collected. Repeated filling of the ZHE to obtain 25 grams of solid is not permitted.

Do not allow the waste, the initial liquid phase, or the extract to be exposed to the atmosphere for any more time than is absolutely necessary. Any manipulation of these materials should be done when cold (4°C) to minimize loss of volatiles.

All glassware and equipment must be thoroughly cleaned before use. A hot soapy water wash and rinse with tap water, then repeat following with a DI water rinse. Rinse with methanol and a final rinse with DI water.

7.3.1 Pre-weigh the (evacuated) filtrate collection container (Step 4.6) and set aside. If using a TEDLAR® bag, express all liquid from the ZHE device into the bag, whether for the initial or final liquid/solid separation, and take an aliquot from the liquid in the bag for analysis.

7.3.2 Place the ZHE piston within the body of the ZHE (it may be helpful first to moisten the piston O-rings slightly with extraction fluid). Adjust the piston within the ZHE body to a height that will minimize the distance the piston will have to move once the ZHE is charged with sample (based upon sample size requirements determined from Section 7.3, Section 7.1.1 and/or 7.1.2). Secure the gas inlet/outlet flange (bottom flange) onto the ZHE body in accordance with the manufacturer's instructions. Secure the glass fiber filter between the support screens and set aside. Set liquid inlet/outlet flange (top flange) aside.

7.3.3 If the sample is 100% solid, weigh out a subsample (25 gram maximum) of the waste, record weight, and proceed to Section 7.3.5.

7.3.4 If the sample contains < 0.5% dry solids, the liquid portion of waste, after filtration, is defined as the 1312 extract. Filter enough of the sample so that the amount of filtered liquid will support all of the volatile analyses required. For samples containing $\geq$ 0.5% dry solids, use the percent solids information obtained in Step 7.1.1 to determine the optimum sample size to charge into the ZHE. The recommended sample size is as follows:

7.3.4.1 For samples containing < 5% solids, weigh out a 500 gram subsample of waste and record the weight.

7.3.4.2 For samples containing >5% solids, determine the amount of waste to charge into the ZHE as follows:

$$\text{Weight of waste to charge ZHE} = \frac{25}{\text{percent wet solids}} \times 100$$

Weigh out a subsample of the waste of the appropriate size and record the weight.

7.3.5 If particle size reduction of the solid portion of the waste was required in Section 7.1.3, proceed to Section 7.3.6. If particle size reduction was not required in Section 7.1.3, proceed to Section 7.3.7.

7.3.6 Prepare the sample for extraction by crushing, cutting, or grinding the solid portion of the waste to a surface area or particle size as described in Section 7.1.3.1. The means used to effect particle size reduction must not generate heat in and of itself. If reduction of the solid phase of the waste is necessary, exposure of the waste to the atmosphere should be avoided to the extent possible.

NOTE: Sieving of the waste is not recommended due to the possibility that volatiles may be lost. The use of an appropriately graduated ruler is recommended as an acceptable alternative. Surface area requirements are meant for filamentous (e.g., paper, cloth) and similar waste materials. Actual measurement of surface area is not recommended.

When the surface area or particle size has been appropriately altered, proceed to Section 7.3.7.

7.3.7 Waste slurries need not be allowed to stand to permit the solid phase to settle. Do not centrifuge wastes prior to filtration.

7.3.8 Quantitatively transfer the entire sample (liquid and solid phases) quickly to the ZHE. Secure the filter and support screens into the top flange of the device and secure the top flange to the ZHE body in accordance with the manufacturer's instructions. Tighten all ZHE fittings and place the device in the vertical position (gas inlet/outlet flange on the bottom). Do not attach the extract collection device to the top plate.

NOTE: If sample material (>1% of original sample weight) has obviously adhered to the container used to transfer the sample to the ZHE, determine the weight of this residue and subtract it from the sample weight determined in Section 7.3.4 to determine the weight of the waste sample that will be filtered.

Attach a gas line to the gas inlet/outlet valve (bottom flange) and, with the liquid inlet/outlet valve (top flange) open, begin applying gentle pressure of 1-10 psi (or more if necessary) to force all headspace slowly out of the ZHE device into a hood. At the first appearance of liquid from the liquid inlet/outlet valve, quickly close the valve and discontinue pressure. If filtration of the waste at 4°C reduces the amount of expressed liquid over what would be expressed at room temperature, then allow the sample to warm up to room temperature in the device before filtering. If the waste is 100% solid (see Section 7.1.1), slowly increase the pressure to a maximum of 50 psig to force most of the headspace out of the device and proceed to Section 7.3.12.

7.3.9 Attach the evacuated pre-weighed filtrate collection container to the liquid inlet/outlet valve and open the valve. Begin applying gentle pressure of 1-10 psig to force the liquid phase of the sample into the filtrate collection container. If no additional liquid has passed through the filter in any 2 minute interval, slowly increase the pressure in 10 psig increments to a maximum of 50 psig. After each incremental increase of 10 psig, if no additional liquid has passed through the filter in any 2 minute interval, proceed to the next 10 psig increment. When liquid flow has ceased such that continued pressure filtration at 50 psig does not result in any additional filtrate within a 2 minute period, stop the filtration. Close the liquid inlet/outlet valve, discontinue pressure to the piston, and disconnect and weigh the filtrate collection container.

NOTE: Instantaneous application of high pressure can degrade the glass fiber filter and may cause premature plugging.

7.3.10 The material in the ZHE is defined as the solid phase of the waste and the filtrate is defined as the liquid phase.

NOTE: Some samples, such as oily wastes and some paint wastes, will obviously contain some material that appears to be a liquid. Even after applying pressure filtration, this material will not filter. If this is the case, the material within the filtration device is defined as a solid and is carried through the 1312 extraction as a solid.

If the original waste contained <0.5% dry solids, this filtrate is defined as the 1312 extract. Proceed to Section 7.3.15.

7.3.11 The liquid phase may now be either analyzed immediately or preserved and stored at 4°C under minimal headspace conditions until time of analysis. Determine the weight of extraction fluid #3 to add to the ZHE as follows:

$$\text{Weight of extraction fluid} = \frac{20 \text{ x percent wet solids x weight of waste filtered}}{100}$$

7.3.12 The following details how to add the appropriate amount of extraction fluid to the solid material within the ZHE and agitation of the ZHE vessel. Extraction fluid #3 is used in all cases.

7.3.12.1 With the ZHE in the vertical position, attach a line from the extraction fluid reservoir to the liquid inlet/outlet valve. The line used shall contain fresh extraction fluid and should be preflushed with fluid to eliminate any air pockets in the line. Release gas pressure on the ZHE piston (from the gas inlet/outlet valve), open the liquid inlet/outlet valve, and begin transferring extraction fluid (by pumping or similar means) into the ZHE. Continue pumping extraction fluid into the ZHE until the appropriate amount of fluid has been introduced into the device.

7.3.12.2 After the extraction fluid has been added, immediately close the liquid inlet/outlet valve and disconnect the extraction fluid line. Check the ZHE to ensure that all valves are in their closed positions. Manually rotate the device in an end-over-end fashion 2 or 3 times. Reposition the ZHE in the vertical position with the liquid inlet/outlet valve on top. Pressurize the ZHE to 5-10 psig (if necessary) and slowly open the liquid inlet/outlet valve to bleed out any headspace that may have been introduced due to the addition of extraction fluid. This bleeding shall be done quickly and shall be stopped at the first appearance of liquid from the valve. Re-pressurize the ZHE with 5-10 psig and check all ZHE fittings to ensure that they are closed.

7.3.12.3 Place the ZHE in the rotary agitation apparatus and rotate at 30 $\pm$ 2 rpm for 18 $\pm$ 2 hours. Ambient temperature (i.e., temperature of room in which extraction occurs) shall be maintained at 23 $\pm$ 2°C during agitation. Record both tumble rate and temperature.

7.3.13 Following the 18 $\pm$ 2 hour period, check the pressure behind the ZHE piston by quickly opening and closing the gas inlet/outlet valve and noting the escape of gas. If the pressure has not been maintained (i.e., no gas release observed), the device is leaking. Check the ZHE for leaking as specified in Section 4.2.1, and perform the extraction again with a new sample of waste. If the pressure within the device has been maintained, the material in the extractor vessel is once again separated into its component liquid and solid phases. If the waste contained an initial liquid phase, the liquid may be filtered directly into the same filtrate collection container (i.e., TEDLAR® bag or VOC vials) holding the initial liquid phase of the waste. A separate filtrate collection container must be used if combining would create multiple phases, or there is not enough volume left within the filtrate collection container. Filter through the glass fiber filter, using the ZHE device as discussed in Section 7.3.9. All extract shall be filtered and collected if the TEDLAR® bag is used, if the extract is multiphasic, or if the waste contained an initial liquid phase.

NOTE: An in-line glass fiber filter may be used to filter the material within the ZHE if it is suspected that the glass fiber filter has been ruptured.

7.3.14 If the original sample contained no initial liquid phase, the filtered liquid material obtained from above is defined as the 1312 extract. If the sample contained an initial liquid phase, the filtered liquid material obtained from above and the initial liquid phase (Section 7.3.9) are collectively defined as the 1312 extract.

7.3.15 Following collection of the 1312 extract, immediately prepare the extract for analysis and store with minimal headspace at 4°C HCI until analyzed. Analyze the 1312 extract according to the appropriate analytical methods. If the individual phases are to be analyzed separately (i.e., are not miscible), determine the volume of the individual phases (to 0.5%), conduct the appropriate analyses, and combine the results mathematically by using a simple volume-weighted average:

$$\text{Final Analyte Concentration} = \frac{(V_1)(C_1) + (V_2)(C_2)}{V_1 + V_2}$$

where:

V_1 = The volume of the first phases (L).
C_1 = The concentration of the analyte of concern in the first phase (mg/L).
V_2 = The volume of the second phase (L).
C_2 = The concentration of the analyte of concern in the second phase (mg/L).

8.0 QUALITY ASSURANCE

8.1 A minimum of one blank (using the same extraction fluid as used for the samples) must be analyzed for every 20 extractions that have been conducted in an extraction vessel. No bias correction is to be taken into consideration (Fr 57, 227).

8.2 A matrix spike shall be performed for each waste type (e.g., wastewater treatment sludge, contaminated soil, etc.) unless the result exceeds the regulatory level and the data is being used solely to demonstrate that the waste property exceeds the regulatory level, the spike concentration may be as low as one half of the analyte concentration, but may not be less than five times the method detection limit. In order to avoid differences in matrix effects, the matrix spikes must be added to the same nominal volume of 1312 extract as that which was analyzed for the unspiked sample.

8.2.1 Matrix spikes are to be added after filtration of the 1312 extract and before preservation. Matrix spikes should not be added prior to 1312 extraction of the sample.

8.2.2 The matrix spikes should be added at a concentration equivalent to the corresponding regulatory level. If the analyte concentration is less than one half the regulatory level, the spike concentration may be as low as one half of the analyte concentration, but may not be less than five times the method detection limit. In order to avoid differences in matrix effects, the matrix spikes must be added to the same nominal volume of 1312 extract as that which was analyzed for the unspiked sample.

8.2.3 The matrix spike is to monitor the performance of the analytical methods used, and to determine whether matrix interferences exist. Use of other internal calibration methods, modification of the analytical methods, or use of alternate analytical methods may be needed to accurately measure the analyte concentration in the 1312 extract when the recovery of the matrix spike is below the expected analytical method performance.

8.2.4 Matrix spike recoveries are calculated by the following formula:

$$\%R\ (\%Recovery) = 100\ (X_s - X_u)/K$$

where:

X_s = measured value for the spiked sample,
X_u = measured value for the unspiked sample, and
K = known value of the spike in the sample.

8.3 All quality control measures described in each analytical methods must be followed.

8.4 The use of internal calibration quantitation methods shall be employed for a metallic contaminant if: (1) Recovery of the contaminant from the 1312 extract is not at least 50% and the concentration does not exceed the appropriate regulatory level, and (2) The concentration of the contaminant measured in the extract is within 20% of the appropriate regulatory level.

8.4.1 The method of standard addition (MSA) shall be employed as the internal calibration quantitation method for each metallic contaminant.

8.4.2 The method of standard additions requires preparing calibration standards in the sample matrix rather than reagent water or blank water or blank solution. It requires taking four identical aliquots of the solution and adding known amounts of standard to three of these aliquots. The forth aliquot is the unknown. Preferably, the first addition should be prepared so that the resulting concentration is approximately 50% of the expected concentration of the sample. The second and third additions should be prepared so that the concentrations are approximately 100% and 150% of the expected concentration of the sample. All four aliquots are maintained at the same final volume by adding reagent water or a blank solution, and may need dilution adjustment to maintain the signals in the linear range of the instrument technique. Analyze all four aliquots.

8.4.3 Prepare a plot, or subject data to linear regression, of instrument signals or external-calibration-derived concentrations as the dependant variable (y-axis) versus concentrations of the additions of standards as the independent variable (x-axis). Solve for the intercept of the abscissa (the independent variable, x-axis) which is the concentration in the unknown.

8.4.4 Alternately, subtract the instrumental signal or external-calibration-derived concentration of the unknown (unspiked) sample from the instrumental signals or external-calibration-derived concentrations of the standard additions. Plot r subject to linear regression of the corrected instrument signals or external-calibration-derived concentrations as the dependant variable versus the independent variable. Derive concentrations for the unknowns using the internal calibration curve as if it were an external calibration curve.

8.5 Samples must undergo 1312 extraction within the following time periods:

SAMPLE MAXIMUM HOLDING TIMES [DAYS]

	From: Field collection To: 1312 extraction	From: 1312 extraction To: Preparative extraction	From: Preparative extraction To: Determinative analysis	Total elapsed time
Volatiles	14	NA	14	28
Semi-volatiles	14	7	40	61
Mercury	28	NA	28	56
Metals, except mercury	180	NA	180	360

NA = Not applicable

If holding times are exceeded, the values obtained will be considered minimal concentrations. Exceeding the holding time is not acceptable in establishing that a waste does not exceed the regulatory level. Of course, exceeding the holding time will not invalidate characterization if the waste exceeds the regulatory level.

9.0 HEALTH AND SAFETY

9.1 Care should be used in handling all samples. Safety glasses must be worn in the lab at all times. The use of blue nitrile gloves and lab coats is highly recommended.

9.2 Research into expected sample content and concentration should be done in order to be prepared for additional safety considerations. Generally, any samples which need special consideration have applicable notes on the sample logs.

9.3 MSDS are available for all reagents and standards, which have been purchased. These are located in the administrative section next to the break room.

9.4 Please see *Waste Disposal; SOP-405* for proper disposal of the waste generated from this area.

EMPIRICAL LABORATORIES, LLC
STANDARD OPERATING PROCEDURE

ORGANICS: SOP 201 REVISION #: 20 EFFECTIVE DATE: 042610

GC/MS SEMIVOLATILES and LOW-CONCENTRATION PAHs BY EPA METHOD 625 AND SW846 METHOD 8270C AND 8270D INCLUDING ADDITIONAL APPENDIX IX COMPOUNDS

APPROVALS:

Lab Director: ____________________ Date: 4/27/10

Data Quality Manager: ____________________ Date: 4/27/10

Section Supervisor: ____________________ Date: 4/27/10

Changes Summary

Revision 20, 4/13/10

- The SOP is an update from Revision 19 dated 4/11/2010
- The SOP is formatted to simplify the text and place all method/program specifications in the SOP tables.

Revision 19, 4/11/10

- The SOP is an update from Revision 18 dated 9/16/08
- The SOP is formatted to include all 22-elements required per the NELAC standards
- The laboratory's revision of all technical SOPs now includes a Table of Contents that provides the map of the technical information contained within the SOP.
- Additional requirements, based upon the DOD QSM 4.1, have been integrated into the routine sample flow; however, if the requirement is different from routine sample flow, then the requirement is outlined and documented as such to be followed only when DOD samples are analyzed.

Table of Contents

1.0 Identification of the Test Method

This SOP is based primarily on SW-846 Methods 8000B/8000C/8270C/8270D. Methods *Federal Register* Method 625 and CLP Method for Semi-volatiles have also been used in the development of this SOP.

2.0 Applicable Matrix or Matrices

This SOP is used for the analysis of semi-volatile organic compounds (including low concentration PAHs) in a variety of matrices (soils, sediments, waters, etc.).

3.0 Detection Limits – Reporting Limits

See Table 1

4.0 Scope of Application, Including Components to Be Analyzed

4.1 Each parameter that is routinely analyzed and reported under the scope of this SOP is listed in the Appendix of this SOP. This table also lists the associated Detection Limit, Limit of Detection and Reporting Limit (also defined as the Limit of Quanititation).

4.2 Extreme care should be taken when working with pure standard and stock standard solutions of these compounds and all handling of standards should be done in a hood. These compounds have been classified as known or suspected human or mammalian carcinogens.

5.0 Summary of the Test Method

5.1 After sample preparation using the appropriate extraction technique, the sample is introduced into the GC/MS using direct injection. The analytes are separated in the gas chromatograph by a combination of the temperature program, the pressure program and the capillary column. The analytes are then detected by the mass spectrometer. Analytes are identified by comparing the mass spectra of known standards with the mass spectra from the sample. Analytes are quantitated relative to known standards using the internal standard method.

6.0 Definitions –

Laboratory Quality System SOP QS08 "Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures" provides information on the commonly used definitions.

7.0 Interferences

7.1 All raw data (samples & QC) must be evaluated for interferences. If contamination occurs, determine whether the source of interference is in the preparation or clean-up of the samples and take corrective action to eliminate the problem.

7.2 Contamination by carryover can occur when samples of high-concentration and low-concentration are analyzed sequentially. To reduce carryover, the sample syringe must be rinsed with solvent between injections. If an unusually high sample is detected, a solvent blank should be analyzed for cross contamination or the subsequent sample should be evaluated for cross-contamination.

8.0 Safety

8.1 Laboratory SOP QS13 "Safety Program & Chemical Hygiene Plan" discusses the safety program that is to be followed lab-wide.

8.2 Care should be used in handling all samples. Safety glasses must be worn in the lab at all times. The use of gloves and lab coats is highly recommended.

8.3 Research into expected sample content and concentration should be done in order to be prepared for additional safety considerations. Generally, any samples which need special consideration have applicable notes on the sample logs.

8.4 MSDS sheets are available for all reagents and standards which have been purchased. These are located on the bookshelf outside the office supply storage room.

9.0 Equipment & Supplies

a HP 5890/6890/7890GC complete with electronic pressure control and temperature programmable gas chromatograph suitable for split-less injection.

b Column: RTX-5MS (or equivalent) 30 m x 0.25 mm I.D. x 0.25 µm film thickness fused silica capillary column.

c HP 5971/5973/5975 mass spectrometer capable of scanning from 35 to 500 amu every second or less, using 70 volts electron energy in electron impact ionization mode. The mass spectrometer is capable of producing a mass spectrum for decafluorotriphenylphosphine, DFTPP, which meets all the tuning criteria of the EPA methods.

d HP 7673/7683 autosampler capable of reproducibility from one injection to another proven by meeting QC and calibration criteria.

e HP GC/MS interface that gives acceptable calibration points at 50 ng per injection for each compound of interest and achieves acceptable tuning performance criteria.

f Acquisition Software: HP Chemstation system is interfaced to the GC/MS. The system acquires and stores data throughout the chromatographic programs.

g Data Processing Software: Target DB on Windows NT server data system is interfaced to the HP Chemstation. The system accepts and stores acquired data. It plots by extracted ion current profile (EICP). The system is also capable of integrating the abundances in any EICP between specified times or scan-number limits.

h Micro syringes – gas tight 5uL and larger.

i Liners – 2mm or 4mm single goose-neck.

j Septa 11mm.

k Seals- dual vespel stainless steel or gold plated 0.8mm.

l Vials- 2ml and larger amber.

m Volumetric flasks- 10ml and larger class A with glass stopper.

10.0 Reagents and Standards –

10.1 The laboratory's LIMS system allows for complete documentation and for the traceability of reagents and standards used within the laboratory.

10.2 Reagent grade chemicals shall be used in all tests unless otherwise specified. All reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

10.3 Methylene chloride (Please read SOP-336 before handling this solvent in our laboratory.) – Trace analysis grade.

10.4 Stock standards are purchased in mixtures from reputable vendors. The date they are received is noted on the label and recorded on the certificate of analysis sheet. The date they are opened is noted on the label and recorded in LIMS. Each standards label is completed with the standard number, name, preparation date, expiration date, solvent and analyst initials. All stocks and standards are stored in the freezer at a temperature of -15°C $\pm$ 5°C from the date they are received/prepared. Standards are brought to room temperature before being used to make standards. Sonication is used if precipitation is observed after bringing to room temperature. The refrigerator and freezer temperature are monitored daily with an annually calibrated thermometer and recorded with calibration correction in the Extraction temperature/calibration logbook.

10.5 Individual standard makeup is recorded in LIMS with specific details concerning the standard being used, concentration, amount, solvent and expiration date.

11.0 Sample Collection, Preservation, Shipment, and Storage

Section 3.0 and table 3-1 of the Empirical Laboratories' Quality Assurance Manual include details concerning sample preservation, containers and handling of semi-volatile samples and extracts. All water and soil samples are stored in the appropriate walk-in coolers at a temperature of 4°C. All extracts are stored in the Hobart in the Extraction lab at a temperature of 4°C. Water samples have a holding time of 7 days from date of sampling while soil samples have a holding time of 14 days from date of sampling (unless otherwise specified for the project). Extracts have 40 days from date of extraction to be analyzed.

12.0 Quality Control

12.1 Internals - All samples and QC are spiked with internal standards prior to analysis.

12.2 Surrogates - All samples and QC are spiked with surrogates prior to extraction. See **Table 2** for criteria and corrective action.

12.3 LCS Sample - The LCS is extracted 1 per extraction batch of up to 20 samples to provide accuracy results. It is spiked using an alternate source or lot number than the calibration standards. See **Table 2** for criteria and corrective action.

12.4 Method Blanks - The Method Blank is extracted 1 per extraction batch of up to 20 samples. See **Table 2** for criteria and corrective action.

12.5 Matrix Spike/Matrix Spike Duplicate (MS/MSD) Sample - 1 in 20 samples are spiked for a MS/MSD, if sample is available. If no sample is available, an LCSD must be extracted to provide precision results. See **Table 2** for criteria and corrective action. Some factors that may affect MS/MSD results are:

12.5.1 Sample matrix - If the sample is a soil, grab sample or sequentially collected water sample it may affect the %R and RPD of the MS/MSD. Corrective action must be taken in the form of reanalysis if a method problem is indicated.

12.5.2 Original sample concentration - If a spiked compound has a problem and the concentration of that compound in the original sample was four or more times the concentration of the spike, no further corrective action may be necessary other than the generation of a corrective action report to document the problem.

12.5.3 MS vs. MSD - If a spiked compound has a similar problem in both the MS and MSD and is not traced to a method problem, no further action may be necessary other than the generation of a non-conformance report to document the problem.

12.5.4 Non-target Interference - The presence of significant non-target interference should be brought to the immediate attention of your supervisor who should discuss the problem with the client/project manager to determine the action to be taken.

12.6 Demonstration of Capability (DOC) – Each new analyst must complete a demonstration of capability by analyzing four LCSs with acceptable precision and accuracy. This also must be done when a new instrument is installed or a significant change to the method has been made.

13.0 Calibration and Standardization

13.1 Quality Systems **SOP QS08 "**Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures" related to Calibration Procedures provides laboratory wide protocols for calibration and standardization.

13.2 Initial Calibration - An initial multi-point calibration curve must be analyzed and shown to meet the initial calibration criteria before any sample analyses may be performed. See **Table 2** for criteria and corrective action. The lowest standard must be less than or equal to the reported quantitation limit and the highest standard must not exceed the linear range of the detector. Generally, levels for the curve range from 1.0ug/mL to 100ug/mL for regular SVOCs and 0.1µg/mL to 50µg/mL for low-concentration PAHs.. Any manual integrations are documented by inclusion of the integrated signals (**before and after manual integration**) initialed, reason indicated and dated with the quantitation report and chromatogram. All integrations are second-checked for acceptability by a senior analyst. Refer to SOP-QS07 for guidance.

13.3 Initial Calibration Verification (ICV) - A second source standard at the continuing calibration verification (CCV) level must be analyzed and calculated against the initial calibration curve, then shown to meet the ICV criteria before any sample analyses may be performed. See **Table 2** for criteria and corrective action. For ICV standard preparation, refer to LIMS. Any manual integrations are documented by inclusion of the integrated signals (**before and after manual integration**) initialed, reason indicated and dated with the quantitation report and chromatogram. All integrations are second-checked for acceptability by a senior analyst. Refer to SOP-QS07 for guidance.

13.4 Continuing Calibration Verification (CCV) - Every 12 hours, a CCV must be analyzed and calculated against the initial calibration curve, then shown to meet the calibration check criteria before any sample analyses may be performed. See **Table 2** for criteria and corrective action. For ICV standard preparation, refer to LIMS. Any manual integrations are documented by inclusion of the integrated signals (**before and after manual integration**) initialed, reason indicated and dated with the quantitation report and chromatogram. All integrations are second-checked for acceptability by a senior analyst. Refer to SOP-QS07 for guidance.

14.0 Procedure

Prior to analysis the samples are prepared for chromatography using the appropriate sample preparation and clean up methods (generally SW-846 methods 3510, 3520, 3541, 3546 3550, 3580, EPA method 625 or CLP).

14.1 Chromatographic conditions: Refer to corresponding instrument maintenance log for current gas chromatograph and mass spectrometer conditions.

14.2 Tuning - Prior to any calibration or analysis, DFTPP tuning criteria must be met for a 50 ng injection of the tuning standard. The injection port performance compounds (pentachlorophenol, benzidine and 4,4'-DDT) are also injected to verify the performance of the injection port . See **Table 2** for criteria and corrective action.

14.3 Extracts - Prior to analysis, 1.0 mL extracts are prepared by verifying volume and spiking with 20uL of the internal standard solution.

14.5 Instrument sequence-The instrument sequence log is filled out prior to sample analyses. An example of a typical instrument sequence log follows:

1-SEQ-TUN1 (12:00 am)
2-SEQ-CCV1
3-SEQ-BS1
4-SEQ-BLK1
5-Sample
6-Sample
7-Sample
8-Sample
9-Sample
10-Sample
11-Sample
12-Sample
13-Sample
14-SEQ-MS1
15-SEQ-MSD1
16-SEQ-TUN2 (12:00pm - 12 hours since last DFTPP/CCV)
17-SEQ-CCV2
18-Sample
19-Sample
20-Sample

14.6 Data Reduction/Evaluation - Each sample analysis sequence is documented using the computer run log generated on the Chemstation. This run log is signed, dated and paginated then placed in a 3 ring binder for that instrument. After the sample has been analyzed, the data is processed through Target DB on the Windows NT data system. The following must be checked to determine if the sample will need reanalysis or dilution. Criteria and corrective action are found in Table 2. Formal data evaluation is detailed in SOP QS05 and documented using the Analyst Data Review Checklist (see Appendix). Manual integration guidance is found in SOP QS07.

14.6.1 Internal Standard Area Counts and Retention Times

14.6.2 Surrogate Recoveries and Retention Times

14.6.3 Analyte concentration.
14.6.4 Analyte identification based on spectrum and retention time.
14.6.5 Analyte quantitation verification.

15.0 Data Analysis and Calculations

15.1 Quality Systems SOP QS09 "General and Commonly used Laboratory Calculations" provides details on general calculations used throughout the laboratory.

15.2 The RF is calculated as follows:

$$RF = \frac{A_s \times C_{is}}{A_{is} \times C_s}$$

where:
A_s = Peak area (or height) of the analyte or surrogate.
A_{is} = Peak area (or height) of the internal standard.
C_s = Concentration of the analyte or surrogate.
C_{is} = Concentration of the internal standard.

15.2 Calibration verification involves the calculation of the percent drift (linear or quadratic) or the percent difference (average) of the instrument response between the initial calibration and each subsequent analysis of the verification standard. Use the equations below to calculate % Drift or % Difference, depending on the calibration procedure used.

$$\% \text{ Drift} = \frac{(\text{Calculated concentration} - \text{Theoretical concentration}) * 100}{\text{Theoretical Concentration}}$$

where:
Calculated concentration is determined from the initial calibration.
Theoretical concentration is the concentration at which the standard was prepared.

$$\% \text{ Difference} = \frac{(\text{CCV RF} - \text{Average RF}) * 100}{\text{Average RF}}$$

where:
CCV RF is the response factor from the analysis of the verification standard
Average RF is the average of the response factors from the initial calibration.

15.3 Concentration in water samples is calculated as follows: [Note: Using the units specified here for these terms will result in a concentration in units of ng/mL, which is equivalent to µg/L.]

$$\text{Concentration } (\mu g/L) = \frac{(A_s)(C_{is})(D)(V_i)}{(A_{is})(\overline{RF})(V_s)(1000)}$$

where:
A_s = Area (or height) of the peak for the analyte in the sample.
A_{is} = Area (or height) of the peak for the internal standard.
C_{is} = Concentration of the internal standard in the volume extracted in ug/L.
D = Dilution factor, if the sample was diluted prior to analysis. If no dilution was made, D = 1. The dilution factor is always dimensionless.
V_i = Volume of the extract injected (µL). The nominal injection volume for samples and calibration standards must be the same.

$\overline{RF}$ = Mean response factor from the initial calibration.

V_s = Volume of the aqueous sample extracted (mL). If units of liters are used for this term, multiply the results by 1000.

The 1000 in the denominator represents the number of μL in 1 mL. If the injection (V_i) is expressed in mL, then the 1000 may be omitted.

15.4 Concentration in non-aqueous samples is calculated as follows: [Note: Using the units specified here for these terms will result in a concentration in units of ng/g, which is equivalent to μg/kg.]

$$\text{Concentration } (\mu\text{g/kg}) = \frac{(A_s)(C_{is})(D)(V_i)}{(A_{is})(\overline{RF})(W_s)(1000)}$$

where: A_s,

A_{is}, C_{is}, D, and $\overline{RF}$ are the same as for aqueous samples, and

W_s = Weight of sample extracted (g). Either a dry weight or wet weight may be used, depending upon the specific application of the data. If units of kilograms are used for this term, multiply the results by 1000.

The 1000 in the denominator represents the number of μL in 1 mL. If the injection (V_i) is expressed in mL, then the 1000 may be omitted.

15.3 Any questions left unanswered by this SOP should be clarified by reading the referenced method. If questions still remain unanswered, check with the Section Manager, Technical Director and/or Data Quality Manager.

16.0 Method Performance

See SOP QS08 and Table 2 for criteria and corrective actions associated to the following method performance items:

16.1 Method Detection Limit Study or Detection Limit Determination
16.2 Limit of Detection Verification
16.3 Limit of Quantitation or Reporting Limit Verification
16.4 Demonstration of Capability (DOC)
16.5 PT Studies

17.0 Pollution Prevention

Quantity of chemicals purchased should be based on expected usage during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

18.0 Data Assessment and Acceptance Criteria for Quality Control Measures

Quality Control SOP QS05, "Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results", provides details on data assessment and acceptance criteria for Quality Control Measures. **Table 2** of this SOP provides information on QC samples, frequency, and the associated criteria specific to the performance of this method.

19.0 Contingencies for Handling out-of-control or unacceptable data

Quality Control SOP QS05, "Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results", provides details on handling out of control data. **Table 2** within this SOP also lists corrective actions associated with the failure of the various QC samples employed for the performance of this method.

20.0 Waste Management

Laboratory SOP QS14 on Waste Handling discusses general guidelines for the appropriate handling of wastes and the laboratory program on waste management.

21.0 References

40 CFR, Part 136; Appendix A
Test Methods for Evaluating Solid Waste, SW-846
National Environmental Laboratory Accreditation Conference; CH. 5, 2003
USACE, EM 200-1-3; Appendix 1; Shell, 2/2001
DOD Quality Systems Manual for Environmental Laboratories,

22.0 Tables, Diagrams, Flowcharts and Validation Data

22.1 Table 1, all applicable parameters with the applicable DL(MDL)/LOD/LOQ(MRL).
22.2 Table 2, QA/QC summary table
22.3 Table 3, Technical Completeness / Accuracy Checklist
22.4 Table 4, Data Reviewers Checklist(s)
22.5 Table 5, 625 QC Limits
22.6 Table 6, Standards Used
22.7 Table 7, INTERNAL STANDARD ASSOCIATION / QUANT MASS – Standard SVOC analysis
22.8 Table 8, LOW CONCENTRATION PAH INTERNAL STANDARD/SURROGATE SPECIFICATIONS
22.9 Figure 1, Tailing Factor Calculation
22.10 Table 9, DFTPP Tuning Criteria

TABLE 1

Analyte (Water)	DL	LOD	MRL/LOQ	Units
1,1'-Biphenyl	1.25	2.50	5.00	ug/L
1,2,4,5-Tetrachlorobenzene	1.25	2.50	5.00	ug/L
1,2,4-Trichlorobenzene	1.25	2.50	5.00	ug/L
1,2-Dichlorobenzene	1.25	2.50	5.00	ug/L
1,3-Dichlorobenzene	1.25	2.50	5.00	ug/L
1,4-Dichlorobenzene	1.25	2.50	5.00	ug/L
2,3,4,6-Tetrachlorophenol	1.25	2.50	5.00	ug/L
2,4,5-Trichlorophenol	1.25	2.50	5.00	ug/L
2,4,6-Trichlorophenol	1.25	2.50	5.00	ug/L
2,4-Dichlorophenol	1.25	2.50	5.00	ug/L
2,4-Dimethylphenol	5.00	10.0	20.0	ug/L
2,4-Dinitrophenol	12.5	25.0	50.0	ug/L
2,4-Dinitrotoluene	1.25	2.50	5.00	ug/L
2,6-Dinitrotoluene	1.25	2.50	5.00	ug/L
2-Chloronaphthalene	1.25	2.50	5.00	ug/L
2-Chlorophenol	1.25	2.50	5.00	ug/L
2-Methylnaphthalene	1.25	2.50	5.00	ug/L
2-Methylphenol	1.25	2.50	5.00	ug/L
2-Nitroaniline	5.00	10.0	20.0	ug/L
2-Nitrophenol	1.25	2.50	5.00	ug/L
3,3´-Dichlorobenzidine	1.25	2.50	5.00	ug/L
3-Nitroaniline	5.00	10.0	20.0	ug/L
4,6-Dinitro-2-methylphenol	5.00	10.0	20.0	ug/L
4-Bromophenyl phenyl ether	1.25	2.50	5.00	ug/L
4-Chloro-3-methylphenol	1.25	2.50	5.00	ug/L
4-Chloroaniline	1.25	2.50	5.00	ug/L
4-Chlorophenyl phenyl ether	1.25	2.50	5.00	ug/L
4-Methylphenol	1.25	2.50	5.00	ug/L
4-Nitroaniline	5.00	10.0	20.0	ug/L
4-Nitrophenol	5.00	10.0	20.0	ug/L
Acenaphthene	1.25	2.50	5.00	ug/L
Acenaphthylene	1.25	2.50	5.00	ug/L
Acetophenone	1.25	2.50	5.00	ug/L
Anthracene	1.25	2.50	5.00	ug/L
Atrazine	1.25	2.50	5.00	ug/L
Benzaldehyde	1.25	2.50	5.00	ug/L
Benzo (a) anthracene	1.25	2.50	5.00	ug/L
Benzo (a) pyrene	1.25	2.50	5.00	ug/L
Benzo (b) fluoranthene	1.25	2.50	5.00	ug/L
Benzo (g,h,i) perylene	1.25	2.50	5.00	ug/L
Benzo (k) fluoranthene	1.25	2.50	5.00	ug/L
Bis(2-chloroethoxy)methane	1.25	2.50	5.00	ug/L
Bis(2-chloroethyl)ether	1.25	2.50	5.00	ug/L
Bis(2-chloroisopropyl)ether	1.25	2.50	5.00	ug/L
Bis(2-ethylhexyl)phthalate	1.25	2.50	5.00	ug/L
Butyl benzyl phthalate	1.25	2.50	5.00	ug/L
Caprolactam	1.25	2.50	5.00	ug/L
Carbazole	1.25	2.50	5.00	ug/L
Chrysene	1.25	2.50	5.00	ug/L
Dibenz (a,h) anthracene	1.25	2.50	5.00	ug/L
Dibenzofuran	1.25	2.50	5.00	ug/L
Diethyl phthalate	1.25	2.50	5.00	ug/L
Dimethylphthalate	1.25	2.50	5.00	ug/L
Di-n-butyl phthalate	1.25	2.50	5.00	ug/L

Table 1 (Continued)

Analyte (Water)	DL	LOD	MRL/LOQ	Units
Di-n-octyl phthalate	1.25	2.50	5.00	ug/L
Fluoranthene	1.25	2.50	5.00	ug/L
Fluorene	1.25	2.50	5.00	ug/L
Hexachlorobenzene	1.25	2.50	5.00	ug/L
Hexachlorobutadiene	1.25	2.50	5.00	ug/L
Hexachlorocyclopentadiene	1.25	2.50	5.00	ug/L
Hexachloroethane	1.25	2.50	5.00	ug/L
Indeno (1,2,3-cd) pyrene	1.25	2.50	5.00	ug/L
Isophorone	1.25	2.50	5.00	ug/L
Naphthalene	1.25	2.50	5.00	ug/L
Nitrobenzene	1.25	2.50	5.00	ug/L
N-Nitrosodi-n-propylamine	1.25	2.50	5.00	ug/L
N-Nitrosodiphenylamine	1.25	2.50	5.00	ug/L
Pentachlorophenol	5.00	10.0	20.0	ug/L
Phenanthrene	1.25	2.50	5.00	ug/L
Phenol	1.25	2.50	5.00	ug/L
Pyrene	1.25	2.50	5.00	ug/L
Analyte (Soil)	**DL**	**LOD**	**MRL/LOQ**	**Units**
1,1'-Biphenyl	83.3	167	333	ug/Kg
1,2,4,5-Tetrachlorobenzene	83.3	167	333	ug/Kg
1,2,4-Trichlorobenzene	83.3	167	333	ug/Kg
1,2-Dichlorobenzene	83.3	167	333	ug/Kg
1,3-Dichlorobenzene	83.3	167	333	ug/Kg
1,4-Dichlorobenzene	83.3	167	333	ug/Kg
2,3,4,6-Tetrachlorophenol	83.3	167	333	ug/Kg
2,4,5-Trichlorophenol	83.3	167	333	ug/Kg
2,4,6-Trichlorophenol	83.3	167	333	ug/Kg
2,4-Dichlorophenol	83.3	167	333	ug/Kg
2,4-Dimethylphenol	333	667	1330	ug/Kg
2,4-Dinitrophenol	833	1670	3330	ug/Kg
2,4-Dinitrotoluene	83.3	167	333	ug/Kg
2,6-Dinitrotoluene	83.3	167	333	ug/Kg
2-Chloronaphthalene	83.3	167	333	ug/Kg
2-Chlorophenol	83.3	167	333	ug/Kg
2-Methylnaphthalene	83.3	167	333	ug/Kg
2-Methylphenol	83.3	167	333	ug/Kg
2-Nitroaniline	333	667	1330	ug/Kg
2-Nitrophenol	83.3	167	333	ug/Kg
3,3´-Dichlorobenzidine	83.3	167	333	ug/Kg
3-Nitroaniline	333	667	1330	ug/Kg
4,6-Dinitro-2-methylphenol	833	1670	3330	ug/Kg
4-Bromophenyl phenyl ether	83.3	167	333	ug/Kg
4-Chloro-3-methylphenol	83.3	167	333	ug/Kg
4-Chloroaniline	83.3	167	333	ug/Kg
4-Chlorophenyl phenyl ether	83.3	167	333	ug/Kg
4-Methylphenol	83.3	167	333	ug/Kg
4-Nitroaniline	333	667	1330	ug/Kg
4-Nitrophenol	333	667	1330	ug/Kg
Acenaphthene	83.3	167	333	ug/Kg
Acenaphthylene	83.3	167	333	ug/Kg
Acetophenone	83.3	167	333	ug/Kg
Anthracene	83.3	167	333	ug/Kg
Atrazine	83.3	167	333	ug/Kg
Benzaldehyde	83.3	167	333	ug/Kg
Benzo (a) anthracene	83.3	167	333	ug/Kg

Table 1 (Continued)

Analyte (Soil)	DL	LOD	MRL/LOQ	Units
Benzo (a) pyrene	83.3	167	333	ug/Kg
Benzo (b) fluoranthene	83.3	167	333	ug/Kg
Benzo (g,h,i) perylene	83.3	167	333	ug/Kg
Benzo (k) fluoranthene	83.3	167	333	ug/Kg
Bis(2-chloroethoxy)methane	83.3	167	333	ug/Kg
Bis(2-chloroethyl)ether	83.3	167	333	ug/Kg
Bis(2-chloroisopropyl)ether	83.3	167	333	ug/Kg
Bis(2-ethylhexyl)phthalate	83.3	167	333	ug/Kg
Butyl benzyl phthalate	83.3	167	333	ug/Kg
Caprolactam	83.3	167	333	ug/Kg
Carbazole	83.3	167	333	ug/Kg
Chrysene	83.3	167	333	ug/Kg
Dibenz (a,h) anthracene	83.3	167	333	ug/Kg
Dibenzofuran	83.3	167	333	ug/Kg
Diethyl phthalate	83.3	167	333	ug/Kg
Dimethylphthalate	83.3	167	333	ug/Kg
Di-n-butyl phthalate	83.3	167	333	ug/Kg
Di-n-octyl phthalate	83.3	167	333	ug/Kg
Fluoranthene	83.3	167	333	ug/Kg
Fluorene	83.3	167	333	ug/Kg
Hexachlorobenzene	83.3	167	333	ug/Kg
Hexachlorobutadiene	83.3	167	333	ug/Kg
Hexachlorocyclopentadiene	83.3	167	333	ug/Kg
Hexachloroethane	83.3	167	333	ug/Kg
Indeno (1,2,3-cd) pyrene	83.3	167	333	ug/Kg
Isophorone	83.3	167	333	ug/Kg
Naphthalene	83.3	167	333	ug/Kg
Nitrobenzene	83.3	167	333	ug/Kg
N-Nitrosodi-n-propylamine	83.3	167	333	ug/Kg
N-Nitrosodiphenylamine	83.3	167	333	ug/Kg
Pentachlorophenol	333	667	1330	ug/Kg
Phenanthrene	83.3	167	333	ug/Kg
Phenol	83.3	167	333	ug/Kg
Pyrene	83.3	167	333	ug/Kg
Analyte Low PAH (Water)	**DL**	**LOD**	**MRL/LOQ**	**Units**
1-Methylnaphthalene	0.0500	0.100	0.200	ug/L
2-Methylnaphthalene	0.0500	0.100	0.200	ug/L
Acenaphthene	0.0500	0.100	0.200	ug/L
Acenaphthylene	0.0500	0.100	0.200	ug/L
Anthracene	0.0500	0.100	0.200	ug/L
Benzo (a) anthracene	0.0500	0.100	0.200	ug/L
Benzo (a) pyrene	0.0500	0.100	0.200	ug/L
Benzo (b) fluoranthene	0.0500	0.100	0.200	ug/L
Benzo (g,h,i) perylene	0.0500	0.100	0.200	ug/L
Benzo (k) fluoranthene	0.0500	0.100	0.200	ug/L
Chrysene	0.0500	0.100	0.200	ug/L
Dibenz (a,h) anthracene	0.0500	0.100	0.200	ug/L
Fluoranthene	0.0500	0.100	0.200	ug/L
Fluorene	0.0500	0.100	0.200	ug/L
Indeno (1,2,3-cd) pyrene	0.0500	0.100	0.200	ug/L
Naphthalene	0.0500	0.100	0.200	ug/L
Phenanthrene	0.0500	0.100	0.200	ug/L
Pyrene	0.0500	0.100	0.200	ug/L
Analyte Low PAH (Soil)	**DL**	**LOD**	**MRL/LOQ**	**Units**
1-Methylnaphthalene	1.67	3.33	6.67	ug/Kg

Table 1 (Continued)

Analyte Low PAH (Soil)	**DL**	**LOD**	**MRL/LOQ**	**Units**
2-Methylnaphthalene	1.67	3.33	6.67	ug/Kg
Acenaphthene	1.67	3.33	6.67	ug/Kg
Acenaphthylene	1.67	3.33	6.67	ug/Kg
Anthracene	1.67	3.33	6.67	ug/Kg
Benzo (a) anthracene	1.67	3.33	6.67	ug/Kg
Benzo (a) pyrene	1.67	3.33	6.67	ug/Kg
Benzo (b) fluoranthene	1.67	3.33	6.67	ug/Kg
Benzo (g,h,i) perylene	1.67	3.33	6.67	ug/Kg
Benzo (k) fluoranthene	1.67	3.33	6.67	ug/Kg
Chrysene	1.67	3.33	6.67	ug/Kg
Dibenz (a,h) anthracene	1.67	3.33	6.67	ug/Kg
Fluoranthene	1.67	3.33	6.67	ug/Kg
Fluorene	1.67	3.33	6.67	ug/Kg
Indeno (1,2,3-cd) pyrene	1.67	3.33	6.67	ug/Kg
Naphthalene	1.67	3.33	6.67	ug/Kg
Phenanthrene	1.67	3.33	6.67	ug/Kg
Pyrene	1.67	3.33	6.67	ug/Kg
Analyte (TCLP)	**DL**	**LOD**	**MRL/LOQ**	**Units**
1,4-Dichlorobenzene	0.00125	0.00250	0.00500	mg/L
2,4,5-Trichlorophenol	0.00125	0.00250	0.00500	mg/L
2,4,6-Trichlorophenol	0.00125	0.00250	0.00500	mg/L
2,4-Dinitrotoluene	0.00125	0.00250	0.00500	mg/L
2-Methylphenol	0.00125	0.00250	0.00500	mg/L
3-Methylphenol	0.00125	0.00250	0.00500	mg/L
4-Methylphenol	0.00125	0.00250	0.00500	mg/L
Hexachlorobenzene	0.00125	0.00250	0.00500	mg/L
Hexachlorobutadiene	0.00125	0.00250	0.00500	mg/L
Hexachloroethane	0.00125	0.00250	0.00500	mg/L
Nitrobenzene	0.00125	0.00250	0.00500	mg/L
Pentachlorophenol	0.0050	0.0100	0.0200	mg/L
Pyridine	0.00125	0.00250	0.00500	mg/L

Table 2. Organic Analysis by Gas Chromatography/Mass Spectrometry (Methods 625/8270)					
QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Demonstrate acceptable analytical capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel, test method, or sample matrix.	QC acceptance criteria published by DoD, if available; otherwise, method-specific criteria.	Recalculate results; locate and fix problem, then rerun demonstration for those analytes that did not meet criteria (see Section C.1.f of DoD QSM 4.1).	NA.	This is a demonstration of analytical ability to generate acceptable precision and bias per the procedure in Appendix C of DoD QSM 4.1. No analysis shall be allowed by analyst until successful demonstration of capability is complete.
MDL determination	Initial method demonstration required for some states – not required for DoD	Refer to SOP QS09.			
LOD determination and verification	Prior to initial analysis then quarterly verification.	See Box D-13 of DoD QSM 4.1			
LOQ establishment and verification	Prior to initial analysis then quarterly verification.	See Box D-14 of DoD QSM 4.1			
Tuning	Prior to ICAL and at the beginning of each 12-hour period.	Refer to table 8 of this SOP.	Retune instrument and verify. Rerun affected samples.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be accepted without a valid tune.
Breakdown check (DDT Method 8270 only)	At the beginning of each 12-hour period, prior to analysis of samples.	Degradation ≤ 20% for DDT. Benzidine and pentachlorophenol should be present at their normal responses, and should not exceed a tailing factor of 2. [Method 625 – benzidine and pentachlorophenol tailing limits are 3 and 5, respectively, when benzidine or acids are target analytes. Benzidine tailing is specific to benzidine analysis and pentachlorophenol tailing is specific to acid analyte analyses according to 625.]	Correct problem then repeat breakdown checks.	Flagging criteria are not appropriate.	No samples shall be run until degradation ≤ 20%. **Not applied when low concentration PAHs are the only target analytes.**

Table 2. Organic Analysis by Gas Chromatography/Mass Spectrometry (Methods 625/8270) (continued)					
QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Minimum five-point initial calibration (ICAL) for all analytes	ICAL prior to sample analysis.	**1.** Average response factor (RF) for SPCCs: SVOCs ≥ 0.050 [2,4-dinitrophenol, hexachlorocyclopentadiene, N-Nitrosodi-n-propylamine, 4-nitrophenol] Note 1: See table 4 of 8270D SPCC analytes and limits. Note 2: ≥ 0.050 for all low-level PAHs **2.** RSD for RFs for CCCs: SVOCs ≤ 30% and one option below: **Option 1:** RSD for each analyte ≤ 15%; [≤ 20% for non-DoD 8270D; or, ≤35% for non-DoD 625] **Option 2:** linear least squares regression r ≥ 0.995 or r^2 ≥ 0.990; [r ≥ 0.990 for non-DoD analyses] **Option 3:** non-linear regression–coefficient of determination (COD) r^2 ≥ 0.990 (6 points shall be used for second order, 7 points shall be used for third order).	Correct problem then repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until ICAL has passed. Calibration may not be forced through the origin for DoD projects.
Second source calibration verification (ICV)	Once after each ICAL.	All project analytes within ± 20% of true value [± 25% for non-DoD 8270C; or, ± 30% for non-DoD 8270D]	Correct problem and verify second source standard. Rerun second source verification. If that fails, correct problem and repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples should be run until calibration has been verified.
Retention time window position establishment for each analyte and surrogate	Once per ICAL.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the sequence CCV is used.	NA.	NA.	

Table 2. Organic Analysis by Gas Chromatography/Mass Spectrometry (Methods 625/8270) (continued)					
QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Evaluation of relative retention times (RRT)	With each sample.	RRT of each target analyte within ± 0.06 RRT units. Note - retention times may be updated based on the CCV to account for minor performance fluctuations or after routine system maintenance (such as column clipping).	Correct problem, then rerun ICAL.	Flagging criteria are not appropriate.	With each sample, the RRT shall be compared with the most recently updated RRT. If the RRT has changed by more than ±0.06 RRT units since the last update, this indicates a significant change in system performance and the laboratory must take appropriate corrective actions as required by the method and rerun the ICAL to reestablish the retention times.
Continuing calibration verification (CCV)	Daily before sample analysis and every 12 hours of analysis time.	**1.** <u>Average RF for SPCCs</u>: SVOCs ≥ 0.050 [2,4-dinitrophenol, hexachlorocyclopentadiene, N-Nitrosodi-n-propylamine, 4-nitrophenol] Note 1: See table 4 of 8270D SPCC analytes and limits. Note 2: ≥ 0.050 for all low-level PAHs **2.** <u>%Difference/Drift for all target compounds and surrogates</u>: SVOCs ≤ 20%D (Note: D = difference when using RFs or drift when using least squares regression or non-linear calibration). [± 20% for CCCs only non-DoD 8270C]	DoD project level approval must be obtained for each of the failed analytes or corrective action must be taken. Correct problem, then rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since last acceptable CCV.	If reanalysis cannot be performed, data should be qualified and explained in the case narrative. Apply qualifier to all results for the specific analyte(s) in all samples since last acceptable CCV. [For non-DoD 8270C, if CCCs exceed, evaluate all analytes for 20%D and qualify as above]	Problem should be corrected. Results should not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed, holding time has been exceeded or client has approved reporting.
Internal standards verification	Every field sample, standard, and QC sample.	Retention time ± 30 seconds from retention time of the midpoint standard in the ICAL or daily CCV; EICP area within -50% to +100% of ICAL midpoint standard or daily CCV.	Inspect mass spectrometer and GC for malfunctions. Reanalysis of samples analyzed while system was malfunctioning is mandatory.	If corrective action fails in field samples, apply qualifier to analytes associated with the non-compliant IS. Flagging criteria are not appropriate for failed standards.	Sample results are not acceptable without a valid IS verification.

Table 2. Organic Analysis by Gas Chromatography/Mass Spectrometry (Methods 625/8270) (continued)					
QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Method blank	One per preparatory batch.	No analytes detected > ½ RL/LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results. For common laboratory contaminants, no analytes detected > RL/LOQ.	Correct problem. If required, reprep and reanalyze method blank and all samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
LCS containing all analytes to be reported, including surrogates	One per preparatory batch.	QC acceptance criteria specified by client or DoD (appendix G), if available. AFCEE 4.0.02 limits are applied for low concentration PAHs as they are not addressed by DoD. Otherwise, use in-house control limits. In-house control limits may not be greater than ± 3 times the standard deviation of the mean LCS recovery. Low concentration PAH limits	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix Spike (MS)	One per preparatory batch per matrix	Use LCS criteria, above.	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply qualifier if acceptance criteria are not met.	For matrix evaluation only. If MS results are outside the LCS limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error.
Matrix spike duplicate (MSD) or sample duplicate	One per preparatory batch per matrix	MSD: For matrix evaluation, use LCS acceptance criteria above. MSD or sample duplicate: RPD ≤ 30% or client specified limit (between MS and MSD or sample and sample duplicate).	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply qualifier if acceptance criteria are not met.	The data shall be evaluated to determine the source of difference.

Table 2. Organic Analysis by Gas Chromatography/Mass Spectrometry (Methods 625/8270) (continued)					
QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Surrogate spike	All field and QC samples.	**Surrogate** / **Water** / **Solid**: Nitrobenzene-d5: 40-110 / 35-100 2-Fluorobiphenyl: 50-110 / 45-105 Terphenyl-d14: 50-135 / 30-125 Phenol-d6: 10-115 / 40-100 2-Fluorophenol: 20-110 / 35-105 2,4,6-Tribromophenol: 40-125 / 35-125 QC acceptance criteria specified by DoD (above) or Client. Low PAH surrogate limits are 14%-129% soil and 34%-167% water. Otherwise, in-house control limits may be used. No limits specified for Method 625.	For QC and field samples, correct problem then reprep and reanalyze all failed samples for failed surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary.	Apply qualifier to all associated analytes if acceptance criteria are not met. For acid surrogate, qualify acid analytes, for base/neutral surrogates, qualify base/neutral analytes.	
Results reported between DL and LOQ	NA.	NA.	NA.	Apply J-flag to all results between DL and LOQ.	

Table 3, Technical Completeness / Accuracy Checklist

1. Were all the QC check elements analyzed – refer to Table 2 of the SOP
2. Were the QC criteria met
3. In cases of failures, was there an NCR written
4. Were all manual integrations signed
5. Were dilution factors applied correctly
6. Was there supervisory or senior-scientist approval for manual integrations on standards and batch QC samples
7. Was the data uploaded into LIMS via direct upload (i.e. datatool) – if yes, then was a cross check subset of the uploaded values performed
8. If the data was entered into LIMS manually, was a check of all entered values performed
9. Was the red marked data in LIMS checked for accuracy and the corresponding hard copy data documented appropriately
10. Were proper data qualifiers applied to the data in LIMS
11. Was the hard copy package checked for completeness to include all data for the sequence such that the data reviewer could reconstruct sample analyses and validate / approve the data

Table 4, Data Reviewers Checklist (Prior to approving data)

1. Does the hard copy raw data (or electronic raw data) package look complete and include all data points
2. Were QA objectives met and for failures were the appropriate actions taken
3. For direct uploads to LIMS, did a subset cross check match the raw data
4. Did all the manual entries into LIMS match the raw data
5. Were there appropriate signatures and documentation on the raw data
6. Were appropriate LIMS flags used
7. Were manual integrations signed
8. Were manual integrations for calibration and QC samples approved by supervisor
9. Were manual calculations verified

ANALYST DATA REVIEW CHECKLIST

Sample Number(s):	
Batch Number(s):	
Method:	8260B/624/8270C/8270D/625 (Circle One)

QA/QC Item	Yes	No	NA	Second Level Review
1. Is the BFB/DFTPP tune performed every 12 hours and is the tuning criteria met?				
Are the RRFs and % RSDs within QC limits for appropriate analytes for the initial calibration? Check the retention times for compounds with the same spectra. Check compounds with different conc.(e.g. m/p-xylene, ketones, etc.).				
3. Was the initial calibration curve verified by a second source calibration standard (ICV) and have criteria been met?				
4. Does the Continuing Calibration Standard (CCV) meet the criteria for the CCCs, SPCCs and/or 20%D for all analytes.				
5. Is the Method Blank run at the desired frequency and is its concentration for target analytes less than the MDLs?				
6. Are the LCS, MS, MSD within control limits and run at the desired frequency?				
7. Are all sample holding times met, analytes within calibration range, IS areas and surrogate recoveries within QC limits?				
8. Were the Method Blank, LCS, MS, MSD and samples uploaded to the LIMS and verified (at least one calculation per batch uploaded)?				

Comments on any "No" response:

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Primary-Level Review: ________________________ Date: ____________________

Second-Level Review: ________________________ Date: ____________________

Table 5 – 625 QC limits

COMPOUND	SPIKE ADDED (ug/L)	SAMPLE CONCENTRATION (ug/L)	LCS CONCENTRATION (ug/L)	LCS % REC #	QC. LIMITS REC.
Acenaphthene	100.00	0.0000	100.00	100	47-145
Acenaphthylene	100.00	0.0000	100.00	100	33-145
Anthracene	100.00	0.0000	100.00	100	27-133
Benzidine	100.00	0.0000	100.00	100	D-110
Benzo(a)anthracene	100.00	0.0000	100.00	100	33-143
Benzo(b)fluoranthene	100.00	0.0000	100.00	100	24-159
Benzo(k)fluoranthene	100.00	0.0000	100.00	100	11-162
Benzo(g,h,i)perylene	100.00	0.0000	100.00	100	D-219
Benzo(a)pyrene	100.00	0.0000	100.00	100	17-163
bis(2-Chloroethoxy)meth	100.00	0.0000	100.00	100	33-184
bis(2-Chloroethyl)ether	100.00	0.0000	100.00	100	12-158
bis(2-Chloroisopropyl)e	100.00	0.0000	100.00	100	36-166
Bis(2-ethylhexyl)phthal	100.00	0.0000	100.00	100	8-158
4-Bromophenyl-phenyleth	100.00	0.0000	100.00	100	53-127
Butylbenzylphthalate	100.00	0.0000	100.00	100	D-152
4-Chloro-3-methylphenol	100.00	0.0000	100.00	100	22-147
2-Chloronaphthalene	100.00	0.0000	100.00	100	60-118
2-Chlorophenol	100.00	0.0000	100.00	100	23-134
4-Chlorophenyl-phenylet	100.00	0.0000	100.00	100	25-158
Chrysene	100.00	0.0000	100.00	100	17-168
Dibenz(a,h)anthracene	100.00	0.0000	100.00	100	D-227
1,2-Dichlorobenzene	100.00	0.0000	100.00	100	32-129
1,3-Dichlorobenzene	100.00	0.0000	100.00	100	D-172
1,4-Dichlorobenzene	100.00	0.0000	100.00	100	20-124
3,3'-Dichlorobenzidine	100.00	0.0000	100.00	100	D-262
2,4-Dichlorophenol	100.00	0.0000	100.00	100	39-135
Diethylphthalate	100.00	0.0000	100.00	100	D-114
2,4-Dimethylphenol	100.00	0.0000	100.00	100	32-119
Dimethylphthalate	100.00	0.0000	100.00	100	D-112
Di-n-butylphthalate	100.00	0.0000	100.00	100	1-118
4,6-Dinitro-2-methylphe	100.00	0.0000	100.00	100	D-181
2,4-Dinitrophenol	100.00	0.0000	100.00	100	D-191
2,4-Dinitrotoluene	100.00	0.0000	100.00	100	39-139
2,6-Dinitrotoluene	100.00	0.0000	100.00	100	50-158
Di-n-octylphthalate	100.00	0.0000	100.00	100	4-146
Fluoranthene	100.00	0.0000	100.00	100	26-137
Fluorene	100.00	0.0000	100.00	100	59-121
Hexachlorobenzene	100.00	0.0000	100.00	100	D-152
Hexachlorobutadiene	100.00	0.0000	100.00	100	24-116
Hexachlorocyclopentadie	100.00	0.0000	100.00	100	15- 70
Hexachloroethane	100.00	0.0000	100.00	100	40-113
Indeno(1,2,3-cd)pyrene	100.00	0.0000	100.00	100	D-171
Isophorone	100.00	0.0000	100.00	100	21-196
Naphthalene	100.00	0.0000	100.00	100	21-133
Nitrobenzene	100.00	0.0000	100.00	100	35-180
2-Nitrophenol	100.00	0.0000	100.00	100	29-182
4-Nitrophenol	100.00	0.0000	100.00	100	D-132
N-Nitroso-di-methylamin	100.00	0.0000	100.00	100	29- 66
N-Nitrosodiphenylamine	100.00	0.0000	100.00	100	23-100
N-Nitroso-di-n-propylam	100.00	0.0000	100.00	100	D-230
Pentachlorophenol	100.00	0.0000	100.00	100	14-176
Phenanthrene	100.00	0.0000	100.00	100	54-120
Phenol	100.00	0.0000	100.00	100	5-112
Pyrene	100.00	0.0000	100.00	100	52-115
1,2,4-Trichlorobenzene	100.00	0.0000	100.00	100	44-142
2,4,6-Trichlorophenol	100.00	0.0000	100.00	100	37-144

Table 6 - BNA STANDARDS USED

base/neutral mix (2000ppm)		**acids mix (2000ppm)**
bis(2-Chloroethyl)ether		2,4-Dinitrophenol
bis(2-Chloroisopropyl)ether		2-Methylphenol
1,3-Dichlorobenzene		4-Methylphenol
1,2-Dichlorobenzene		Benzoic acid
1,4-Dichlorobenzene		4,6-Dinitro-2-methylphenol
Hexachloroethane		4-Nitrophenol
N-Nitroso-di-methylamine		2,4,5-Trichlorophenol
N-Nitroso-di-n-propylamine		2,4,6-Trichlorophenol
2,4-Dinitrotoluene		Phenol
2,6-Dinitrotoluene		Pentachlorophenol
Fluorene		2-Nitrophenol
Dimethylphthalate		4-Chloro-3-methylphenol
Hexachlorocyclopentadiene		2,4-Dichlorophenol
Anthracene		2,4-Dimethylphenol
4-Bromophenyl-phenylether		Benzoic acid
Di-n-butylphthalate		
bis(2-Chloroethoxy)methane		
1,2-Diphenylhydrazine		**semivoa misc. mix(2000ppm)**
Fluoranthene		Aniline
Hexachlorobenzene		Benzyl alcohol
N-Nitrosodiphenylamine		Carbazole
Phenanthrene		4-Chloroaniline
Hexachlorobutadiene		Dibenzofuran
Isophorone		2-Methylnaphthalene
Naphthalene		2-Nitroaniline
Nitrobenzene		3-Nitroaniline
1,2,4-Trichlorobenzene		4-Nitroaniline
Acenaphthene		Pyridine
Acenaphthylene		
2-Chloronaphthalene		
4-Chlorophenyl-phenylether		**Benzidine mix (2000ppm)**
Diethylphthalate		Benzidine
Benzo(a)anthracene		3,3'-Dichlorobenzidine
Bis(2-ethylhexyl)phthalate		
Butylbenzylphthalate		
Chrysene		**Individual or misc. mixes (2000/5000/20,000ppm)**
p-(Dimethylamino)azobenzene		Caprolactam
Pyrene		Benzaldehyde
Benzo(b)fluoranthene		Atrazine
Benzo(k)fluoranthene		1,1'-Biphenyl
Benzo(g,h,i)perylene		1,4-Dioxane
Benzo(a)pyrene		1-methylnapthalene
Dibenz(a,h)anthracene		2,6-dichlorophenol
Di-n-octylphthalate		2,3,4,6-tetrachlorophenol
Indeno(1,2,3-cd)pyrene		

BNA internals (2000ppm)		Acid surrogate (7500ppm)
1,4-Dichlorobenzene-d4 (I.S)(1)		2-Fluorophenol (S)
Naphthalene-d8 (I.S)(35)		Phenol-d6 (S)
Acenaphthene-d10 (I.S) (59)		2,4,6-Tribromophenol (S)
Phenanthrene-d10 (I.S) (79)		2,-Chlorophenol-d4 (S)
Chrysene-d12 (I.S) (92))		BN surrogate (5000ppm)
Perylene-d12 (I.S) (101)		Nitrobenzene-d5 (S)
		Terphenyl-d14 (S)
		2-Fluorobiphenyl (S)
		1,2-Dichlorobenzene-d4 (S)

Table 7 INTERNAL STANDARD ASSOCIATION / QUANT MASS – Standard SVOC analysis					
COMPOUND	***I.S**	**Q.M**	**COMPOUND**	***I.S**	**Q.M**
1,4-Dichlorobenzene-d4 **(I.S)(1)**		152	Dimethylphthalate	59	163
Acetophenone	1	105	Hexachlorocyclopentadiene	59	237
Aniline	1	93	2,4-Dinitrophenol	59	184
Benzaldehyde	1	106	2,4-Dinitrotoluene	59	165
Benzyl alcohol	1	108	2,6-Dinitrotoluene	59	165
bis(2-Chloroethyl)ether	1	93	Fluorene	59	166
bis(2-Chloroisopropyl)ether	1	45	2-Nitroaniline	59	65
1,3-Dichlorobenzene	1	146	3-Nitroaniline	59	138
1,2-Dichlorobenzene	1	146	4-Nitroaniline	59	138
1,4-Dichlorobenzene	1	146	4-Nitrophenol	59	65
2-Methylphenol	1	108	2,4,5-Trichlorophenol	59	196
4-Methylphenol	1	108	2,4,6-Trichlorophenol	59	196
3-Methylphenol	1	108	2-Fluorobiphenyl (S)	59	172
Phenol	1	94	Phenanthrene-d10 **(I.S) (79)**		188
Pyridine	1	79	Anthracene	79	178
Hexachloroethane	1	117	Atrazine	79	200
N-Nitroso-di-methylamine	1	42	4-Bromophenyl-phenylether	79	248
N-Nitroso-di-n-propylamine	1	70	Carbazole	79	167
2-Fluorophenol (S)	1	112	Di-n-butylphthalate	79	149
Phenol-d6 (S)	1	99	4,6-Dinitro-2-methylphenol	79	198
Naphthalene-d8 **(I.S)(35)**		136	1,2-Diphenylhydrazine	79	77
Benzoic acid	35	105	Fluoranthene	79	202
bis(2-Chloroethoxy)methane	35	93	Hexachlorobenzene	79	284
Caprolactam	35	113	N-Nitrosodiphenylamine	79	169
4-Chloroaniline	35	127	Pentachlorophenol	79	266
4-Chloro-3-methylphenol	35	107	Phenanthrene	79	178
2,4-Dichlorophenol	35	162	2,4,6-Tribromophenol (S)	79	330
2,4-Dimethylphenol	35	107	Chrysene-d12 **(I.S) (92)**		240
Hexachlorobutadiene	35	225	Benzidine	92	184
Isophorone	35	82	Benzo(a)anthracene	92	228
2-Methylnaphthalene	35	141	Bis(2-ethylhexyl)phthalate	92	149
Naphthalene	35	128	Butylbenzylphthalate	92	149
Nitrobenzene	35	77	Chrysene	92	228
2-Nitrophenol	35	139	3,3'-Dichlorobenzidine	92	252
1,2,4-Trichlorobenzene	35	180	p-(Dimethylamino)azobenzene	92	225
Catechol	35	110	Pyrene	92	202
Nitrobenzene-d5 (S)	35	82	Terphenyl-d14 (S)	92	244
Acenaphthene-d10 **(I.S) (59)**		164	Perylene-d12 **(I.S) (101)**		264
Acenaphthene	59	153	Benzo(b)fluoranthene	101	252
Acenaphthylene	59	152	Benzo(k)fluoranthene	101	252
1,1'-Biphenyl	59	154	Benzo(g,h,i)perylene	101	276
2-Chloronaphthalene	59	162	Benzo(a)pyrene	101	252
4-Chlorophenyl-phenylether	59	204	Dibenz(a,h)anthracene	101	278
Dibenzofuran	59	168	Di-n-octylphthalate	101	149
Diethylphthalate	59	149	Indeno(1,2,3-cd)pyrene	101	276
I.S=internal standard, Q.M=quant mass, S=surrogate					

Table 7 INTERNAL STANDARD ASSOCIATION / QUANT MASS – Standard SVOC analysis (contd)

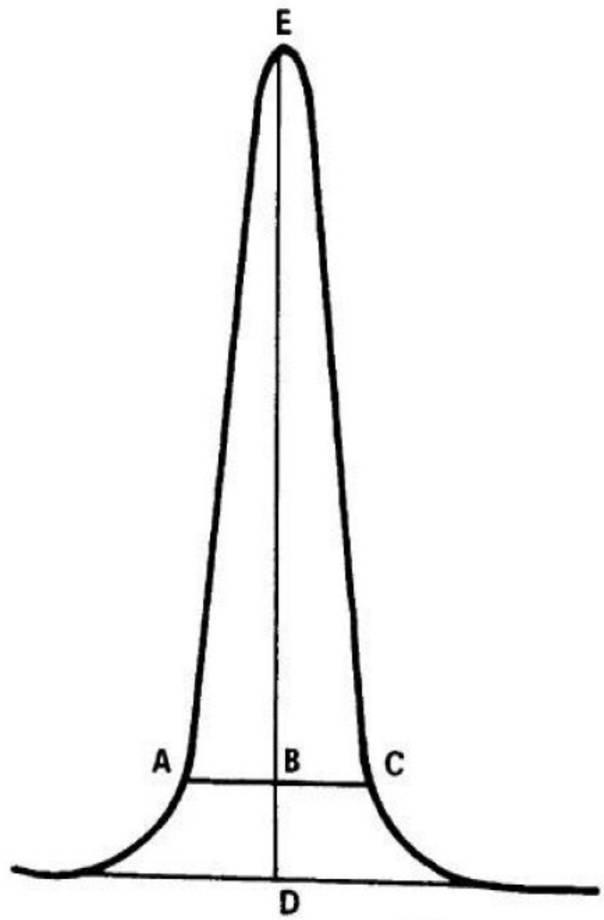
COMPOUND	*I.S	Q.M	COMPOUND	*I.S	Q.M
1,4-Dichlorobenzene-d4 **(I.S)(1)**		152	Diphenylamine	59	169
Pentachloroethane	1	167	Thionazin	59	107
2-Picoline	1	93		59	
N-Nitrosomethylethylamine	1	88		59	
Methyl methanesulfonate	1	80		59	
N-Nitrosodiethylamine	1	102		59	
Ethyl methanesulfonate	1	79		59	
N-Nitrosopyrrolodine	1	100		59	
N-Nitrosomorpholine	1	56		59	
0-Toluidine	1	106		59	
	1		Phenanthrene-d10 **(I.S) (79)**		188
	1		4-Nitroquinoline-1-oxide	79	190
	1		Phenacetin	79	108
	1		4-Aminobiphenyl	79	169
	1		Pentachloronitrobenzene	79	237
	1		Sulfotepp	79	97
	1		Phorate	79	75
Naphthalene-d8 **(I.S)(35)**		136	Diallate	79	86
1- Methylnaphthalene	35	141	Dimethoate	79	87
N-Nitrosopiperidine	35	114	Pronamide	79	173
a,a-Dimethylphenethylamine	35	58	Disulfoton	79	88
O,O,O-Triethylphosphorothioate	35	97	Dinoseb	79	211
Hexachloropropene	35	213		79	
2,6-Dichlorophenol	35	162		79	
p-Phenylenediamine	35	108	Chrysene-d12 **(I.S) (92)**		240
N-Nitrosodi-n-butylamine	35	84	Methapyrilene	92	97
Safrole	35	162	p-(Dimethylamino)azobenzene	92	225
1,2,4,5-Tetrachlorobenzene	35	216	Chlorobenzilate	92	251
	35		3,3'- Dimethylbenzidine	92	212
	35		2- Acetylaminofluorene	92	181
	35		7,12-Dimethylbenz[a]anthracene	92	256
	35		Aramite	92	185
	35		Methyl parathion	92	109
	35		Parathion	92	109
Acenaphthene-d10 **(I.S) (59)**		164	Isodrin	92	193
Isosafrole	59	162	Kepone	92	272
1,4-Naphthoquinone	59	158	Famphur	92	218
Pentachlorobenzene	59	250	Perylene-d12 **(I.S) (101)**	101	
2-Naphthylamine	59	143	3-Methylcholanthrene	101	268
1-Naphthylamine	59	143	Hexachlorophene	101	196
2,3,4,6-Tetrachlorophenol	59	232		101	
5-Nitro-o-toluidine	59	152		101	
I.S=internal standard, Q.M=quant mass, S=surrogate					

Table 8: LOW CONCENTRATION PAH INTERNAL STANDARD/SURROGATE SPECIFICATIONS

INTERNAL STD ASSOCIATION

Phenanthrene-d10 (IS)
Naphthalene
2-Methylnaphthalene
1-Methylnaphthalene
2-Fluorobiphenyl(SUR)
Acenaphthylene
Acenaphthene
Fluorene
Phenanthrene
Anthracene
Fluoranthene
Pyrene
Perylene-d12 (IS)
Terphenyl-d14(SUR)
Benzo(a)anthracene
Chrysene
Benzo(b)fluoranthene
Benzo(k)fluoranthene
Benzo(a)pyrene
Indeno(1,2,3-cd)pyrene
Dibenz(a,h)anthracene
Benzo(g,h,i)perylene

FIGURE 1
TAILING FACTOR CALCULATION



TAILING FACTOR= $\frac{BC}{AB}$

Example calculation: Peak Height = DE = 100 mm
10% Peak Height = BD = 10 mm
Peak Width at 10% Peak Height = AC = 23 mm
AB = 11 mm
BC = 12 mm

Therefore: Tailing Factor = $\frac{12}{11}$ = 1.1

Table 9, DFTPP Tuning Criteria

Mass	Ion Abundance Criteria
51	30-60% of mass 198
68	<2% of mass 69
70	<2% of mass 69
127	40-60% of mass 198
197	<1% of mass 198
198	Base peak, 100% relative
199	5-9% of mass 198
275	10-30% of mass 198
365	>1% of mass 198
441	Present, but less than mass 443
442	>40% of mass 198
443	17-23% of mass 442

Note: While 8270D table 3 indicates different criteria, section 11.3.1.2 allows the use of alternate criteria.

EMPIRICAL LABORATORIES, LLC
STANDARD OPERATING PROCEDURE

ORGANICS: SOP 202 **REVISION #: 22** **EFFECTIVE DATE: 093009**

GC/MS VOLATILES BY EPA METHOD E624 & SW846 METHOD 8260B INCLUDING APPENDIX IX COMPOUNDS

APPROVALS:

Lab Director: ______________ Date: 10/5/09

Data Quality Manager: Marcia ______________ Date: 10/5/09

Section Supervisor: ______________ Date: 10/7/09

Changes Summary

Revision 22, 9/30/09

- The SOP is an update from Revision 21 dated 09/11/08
- The SOP is formatted to include all 22-elements required per the NELAC standards
- The laboratory's revision of all technical SOPs now includes a Table of Contents that provides the map of the technical information contained within the SOP.
- Additional requirements, based upon the DoD QSM 4.1, have been integrated into the routine sample flow; however, if the requirement is different from routine sample flow, then the requirement is outlined and documented as such to be followed only when DoD samples are analyzed.

Table of Contents

1. Identification of the Test Method

1.1 This SOP is compliant with methods – EPA Method 624 and SW-846 Method 8260B

2. Applicable Matrix or Matrices

2.1 This SOP is applicable to – The analysis of volatile organic compounds in a variety of matrices including but not limited to soils, sediments, ground and surface waters, aqueous sludge, oily wastes, etc.

3. Detection Limit: See **Table 1** of this SOP.

4. Scope of Application, Including components to be Analyzed

4.1 This SOP is based primarily on SW-846 Method 8260B. Methods SW-846 Method 8000B; *Federal Register* Method 624; and CLP Method for Volatiles have also been used in the development of this SOP. The analyses by these various methods are clearly defined in the respective regulatory manuals. A good understanding of these different methods is essential to the performance of each method. Each parameter that is analyzed and reported under the scope of this SOP is listed in **Table 1** of this SOP. This table also lists the associated Reporting Limit (also defined as the LOD) and the lowest Calibration level for each analyte. When applicable, surrogate and Internal Standard Analytes are listed and indicated as such within this table.

5. Summary of the Test Method

5.1 After sample preparation, the sample is introduced into the GC/MS generally using purge and trap but sometimes using direct injection (see SW-846 Methods 5030B, 5035 and 3585 for preparation). In purge and trap, the analytes are stripped from the sample using helium and trapped on an adsorbent tube. The tube is heated while being backflushed with helium to carry the analytes to the GC/MS system. The analytes are separated in the gas chromatograph by a combination of the temperature program and the capillary column. The analytes are then detected by the mass spectrometer. Analytes are identified by comparing the mass spectra of known standards with the mass spectra of the sample. Analytes are quantitated relative to known standards using the internal standard method.

6. Definitions

6.1 Laboratory Quality System SOP QS08 "Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures" provides information on the commonly used definitions.

6.2 Additional definitions specific to this SOP are listed below:

amu	atomic mass unit
BFB	Bromofluorobenzene

°C	degrees Centigrade
CLP	Contract Laboratory Program
DOD	Department of Defense
EICP	extracted ion current profile
G	gram or grams
GC/MS	Gas Chromatograph/Mass Spectrometer
I.D.	inner diameter
ISTD	internal standard
µm	micometer
µL	microliter
mL	milliliter
mm	millimeter
ng	nanogram
P&T	purge and trap
SURR	surrogate
SPCC	System Performance Check Compound
TCLP	Toxicity Characteristic Leaching Procedure
USACE	United States Army Corps Of Engineers
VOA	volatile organic analysis

7. Interferences

7.1 Section 3.0 of SW-846 Method 8260B details interferences and potential problems which may be encountered when dealing with volatile analyses.

8. Safety

8.1 Laboratory SOP QS13 "Safety Program & Chemical Hygiene Plan" discusses the safety program that is to be followed labwide.

9. Equipment & Supplies

9.1 GC : HP 5890 or 6890, temperature programmable, suitable for split or splitless injection.

9.2 Column: DB-VRX 60 meter x 0.25 mm I.D. 1.4 µm film thickness or 20 meter x 0.18 mm ID 1.0 µm film thickness silicon coated fused silica capillary column or equivalent.

9.3 M.S. : HP 5971, 5972 or 5973 capable of scanning 35 to 500 amu every one second or less, using 70 volts electron energy in electron impact ionization mode. The MS is capable of producing a mass spectrum for p-Bromofluorobenzene, BFB, which meets all tuning criteria for EPA methods [when 1 µL (50 ng) of the GC/MS tuning standard is introduced to the GC.]

9.4 Purge and Trap Unit

9.4.1 Concentrators: Tekmar LSC 2000 or Tekmar/Dohrmann 3000/3100 Sample Concentrator equipped with Supelco trap number 2-1066-U or 2-4920-U VOCARB 3000 providing good delivery for all target compounds.

9.4.2 Autosamplers: Varian Archon 51 position programmable autosampler with 5ml to 25ml water and heated soil capability.

9.5 Acquisition Software: HP chemstation system interfaced to the GC/MS. The system acquires and stores data throughout the chromatographic programs.

9.6 Data Processing Software: TargetDB on Windows NT data system interfaced to the HP Chemstation. The system accepts and stores acquired data. It plots by extracted ion current profile (EICP). The system is also capable of integrating the abundances of any EICP between specified time or scan-number limits. NBS75K mass spectral library is installed.

9.7 Microsyringes – 1.0, 5.0,10, 25, 100, 250, 500 and 1000 µL.

9.8 Syringes – 5, 25 and 50 mL, gas-tight with Luer end.

9.9 Balance - analytical, 0.0001 g; top-loading, 0.01 g.

9.10 Disposable pasteur pipets.

9.11 Volumetric flasks, Class A - 2 mL, 5 mL, 10 mL, 50 mL, 100 mL and 250 mL with ground-glass stoppers.

9.12 Spatula - stainless steel.

9.13 Glass scintillation vials - 20mL with screw caps.

9.14 Nitrile Gloves

9.15 pH paper (measures pH from 0-14).

10. Reagents and Standards

10.1 The laboratory's LIMS system allows for complete documentation and for the traceability of reagents and standards used within the laboratory. The following information relates to the specific reagents and standards used for the performance of the method:

10.2 Organic-free reagent water - obtained from a modulab system.

10.3 Methanol - Purge and trap grade (EM-Omnisolv EM-0482-6 or equivalent)

10.4 Methanol - suitable for use in gas chromatography (B&J Omnisolv MX0484- 1, or equivalent)

10.5 Sodium bisulfate, $NaHSO_4$ – ACS reagent grade, or equivalent. Available from Aldrich (Part No. 30,782-3).

10.6 Stock standards are purchased in mixtures from reputable vendors. The date they are received is noted on the label. The date they are opened is noted on the label and recorded in the LIMS system along with their lot number and vendor and given a sequential number. Each standard label is completed with the standard number, name, preparation date, expiration date, solvent and analyst initials. Stock standards, when opened, have an expiration date of 6 months, **except for gas standards for South Carolina samples which have a one week expiration date**. All stocks and standards are stored in the freezer at a temperature of -15°C ± 5°C or less from the date they are received/prepared. The freezer temperature is monitored daily with an annually calibrated thermometer and recorded with calibration correction in the VOA refrigerator/freezer logbook. Makeup of common standards is detailed below. See standard ID in LIMS system for makeup of other standards.

10.6.1 The Bromofluorobenzene (BFB) tuning standard is prepared as follows: Using a 50µL syringe, 40µL of standard (BFB @ 2500ng/µL) is injected into a 2mL volumetric flask containing approximately 1.0mL P&T methanol (Vendor,Lot) and diluted to volume with same making a 50ng/µL standard. After capping and inverting 3 times, the solution is transferred to a labeled 2ml, teflon-lined, screw-capped vial and stored in the freezer at -15°C ± 5°C or less for up to 6 months **(1 week for South Carolina samples)**. A direct injection of 1µL (or equivalent purge) is used to tune the instrument.

10.6.2 The internal and surrogate standards are prepared as follows: Using the indicated syringe, the indicated amount of standard is injected into a 50 mL volumetric flask containing P&T methanol (Vendor,Lot) and diluted to volume with same making a 150ng/µL standard. After capping and inverting 3 times, the solution is transferred to the Archon standard vial and stored under helium for 1 month or less. Each 8260/624 sample is automatically injected with 1µL of this standard. The internal standard/surrogate solution will be replaced if the –50%-200% criteria fails in the CCV when calculated against the previous CCV.

Standard	Conc. (ng/µL)	Syringe (µL)	Amount (µL)
8260 ISTD Mix	2500	1000	3000
Surr. Mix	2500	1000	3000

10.6.3 Calibration standards are prepared from the vendor stock standards at appropriate concentrations as follows. Occasionally unusual compounds are added to the mix so it is best to check the VOA standards log book for exact standard makeup. Note: for laboratory control spikes (LCS), alternate sources or lot numbers from the main calibration standard are used to make the LCS standard. See the appendix for analytes in the main mixes.

10.6.3.1 Primary Standard: Using the indicated syringe, the indicated amount of standard is injected into a 2mL volumetric flask containing approximately 1.0mL P&T methanol (Vendor,Lot) and diluted to volume with same to make a 100-500ng/µL standard. After capping and inverting 3 times, the solution is transferred into 2ml amber vial w/mini-inert valve and stored in the freezer at -15°C ± 5°C for 1 week. A 50µg/L (5mL purge) standard is made using 25µL of this standard to 50mLof reagent water.

Stock Standard(CCV)	Conc (ng/µL)	Syringe(µL)	Amount(µL)	Final Conc (ng/µL)
2-CEVE **(Cat#30265)**	20000	25	20	200
Vinyl Acetate **(#3766)**	5000	100	80	200
Ketones **(cat#30006)**	5000	100	80	200
Liquid mix **(C-349H-07)**	2000	100	100	100
Custom mix **(CCS-1037)**	5000	50	40	100
Gases **(cat#30042)**	2000	100	100	100
Acrolein/Acrylonitrile **(CC2098.10)**	20,000	50	50	500

Additional compounds may be added such as Appendix IX. Refer to standard ID in LIMS system.

10.6.4 ICV/LCS/Matrix Spike Mix: A second source standard is used to check the validity of the gas and primary calibration standards used in analyzing the calibration curve. Using the indicated syringe, the indicated amount of standard is injected into a 2mL volumetric flask containing approximately 1.0mL P&T methanol (Vendor,Lot) and diluted to volume with same to make a 100-500ng/µL standard. After capping and inverting 3 times, the solution is transferred into 2ml amber vial w/mini-inert valve and stored in the freezer at -15°C ± 5°C for 1 week. A 50µg/L ICV/LCS/Matrix Spike is made using 25µL of this standard to 50mL of reagent water/Sample Matrix.

Stock Standard(ICV/LCS)	Conc (ng/µL)	Syringe(µL)	Amount(µL)	Final Conc (ng/µL)
2-CEVE	20,000	25	20	200
Vinyl Acetate	5000	100	80	200
Ketones	5000	100	80	200
Liquid mix	2000	100	100	100
Custom Mix	5000	50	40	100
Gases	2000	100	100	100
Acrolein/Acrylonitrile	50,000	50	50	500

11. Sample Collection, Preservation, Shipment, and Storage

11.1 Quality Systems SOP QS10 related to Sample Receipt, Handling, & Processing provides details for collection, preservation, shipment, and storage. All water samples are stored in the BlueIce refrigerator in the VOA lab at a temperature of 4°C. All unpreserved soil samples in TerraCore or encores are stored in the freezer in the VOA lab. All soil samples in bulk jars or chemically preserved TerraCore are stored in the soil walk-in refrigerator at a temperature of 4°C. Non-preserved water volatile samples have a holding time of 7 days from date of sampling. Preserved water samples and soil volatile samples have a holding time of 14 days from date of sampling (unless otherwise specified for the project).

12. Quality Control

12.1 Quality Systems SOP QS08 “Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures” outlines details related to laboratory wide protocols on quality control.

12.1 Internal Standards - All samples and QC are spiked with internals. See **Table 2** for criteria and corrective action.

12.2 Surrogates - All samples and QC are spiked with surrogates. The surrogate recoveries from method blanks and LCS are used to generate control limits. See section 14.5.2 of this SOP for criteria and corrective action. **When analyzing samples for DOD QSM Version 4.1, DOD limits will be used.**

12.3 LCS Sample - An LCS is analyzed every 12 hour tune. To prepare the LCS, a blank is spiked with standards prepared from an alternate vendor or lot number from the calibration standards. Note: the concentration of the LCS will be 20 µg/L when analyzing 624 samples (QC Check Sample). The recoveries are used to generate control limits. The limits are in-house generated matrix spike limits or client specified limits for matrix spike analytes and 70-130% (or client specified limits) recovery for waters or soils for all other analytes if limits have not been generated. Limits for 624 LCSs are taken from table 5 of method 624. If the LCS compound has a recovery above the upper limit, but the same compound is not detected in any of the batch samples, no corrective action is required. For all other situations, the LCS should be reanalyzed for the failed analytes only. If the second analysis fails, all associated samples should be reanalyzed for the failed analytes only. **When analyzing samples for DOD QSM Version 4.1, DOD limits will be used. South Carolina limits are 70-130% except for poor purgers which are 60-140%.**

12.4 Method Blanks - The concentration for method target analytes must be < ½ the Reporting Limit (also defined as the Limit of Quantitation). The first step of corrective action is to assess the affect on the samples. If an analyte is found only in the method blank, but not in any batch samples, no further corrective action may be necessary. Steps should be taken to find/reduce/eliminate the source of this contamination in the method blank. If an analyte is found in the method blank and some, or all, of the other batch samples, then corrective action is required. The source of contamination must be investigated and appropriate action taken and documented to find/reduce/eliminate the source of this contamination. The method blank, and any samples containing the same contaminant, would likely be reanalyzed. For the common laboratory contaminants, meeting the above requirements is not practical. Random cases of contamination are difficult to control, however, daily contamination is not acceptable and corrective action is essential. If a contaminant is found in the method blank and the samples, the compound concentration must be flagged with a 'B' on the final report unless the concentration is greater than 10x that found in the method blank. A method blank is analyzed every 12 hour tune.

12.5 Matrix Spike/Matrix Spike Duplicate (MS/MSD) Sample - 1 in 20 samples are spiked for an MS/MSD with the LCS standard. Criteria for the MS/MSD recoveries are the same as the LCS limits. Limits for the RPDs are 30% RPD for water and soil.. Samples that do not meet these criteria due to matrix must be flagged on the final report for QC problems. Generally, batch control is not based on MS/MSD results unless general method failure is determined to be the problem. In that case, the samples and associated QC would be reanalyzed for the failed analytes only. MS data evaluation must include the consideration of the following factors. **When analyzing samples for DOD QSM Version 4.1, DOD limits will be used.**

12.5.1 Sample matrix - If the sample is a soil, grab sample or sequentially collected water sample it may affect the %R and RPD of the MS/MSD. A water sample which was taken from the same VOA vial for the original sample and the MS/MSD should have very good RPDs unless there has been a method problem. Corrective action must be taken in the form of reanalysis if a method problem is indicated.

12.5.2 Original sample concentration - If a spiked compound has a problem and the concentration of that compound in the original sample was four or more times the concentration of the spike, no further corrective action may be necessary other than the generation of a corrective action report to document the problem.

12.5.3 MS vs. MSD - If a spiked compound has a problem in both the MS and MSD, review the LCS and if acceptable no further action may be necessary since it is attributable to matrix effect.

12.5.4 Non-target Interference - The presence of significant non-target interference should be brought to the immediate attention of your supervisor who should discuss the problem with the client/project manager to determine the action to be taken.

13. Calibration and Standardization

13.1 Quality Systems **SOP QS08 "**Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures" related to Calibration Procedures provides laboratory wide protocols for calibration and standardization.

13.2 Chromatographic conditions – Refer to corresponding instrument maintenance log for current gas chromatograph, mass spectrometer, and concentrator conditions.

13.3 System Bakeout - Prior to analysis an instrument blank is analyzed.

NOTE: Further cleaning may be accomplished by backflushing the lines with methanol and then analyzing blanks overnight.

13.4 Tuning - Prior to any calibration or analysis, BFB tuning criteria must be met for a 1.0µL injection of the tuning standard [see below]. Tune must be met every 12 hours sample analysis is to be performed **(every 24 hours for *Federal Register* Method 624 except for South Carolina which only allows 12 hours).** The mass spectrum of BFB is acquired as follows: by using the BFB method in Target (which uses three scans with backround subtraction) to process the BFB data file. If the BFB tune does not pass criteria corrective action should be taken

m/z	Required Intensity (relative abundance)
50	15 to 40% of m/z 95
75	30 to 60% of m/z 95
95	Base peak, 100% relative abundance
96	5 to 9% of m/z 95
173	Less than 2% of m/z 174
174	Greater than 50% of m/z 95
175	5 to 9% of m/z 174
176	Greater than 95% but less than 101% of m/z 174
177	5 to 9% of m/z 176

13.5 **Calibration:** Calibration standards are made up in water using the appropriate amount of the methanol standard. **Calibration for soils for South Carolina requires that 5mL of sodium bisulfate solution is added to each calibration standard made if the samples will be preserved with sodium bisulfate.** All calibration standard manual integrations must be approved by for acceptability.

13.5.1 Initial Calibration - An initial calibration curve at no less than five (six if using a quadratic curve fit) concentration levels must be analyzed and shown to meet the initial calibration criteria before any sample analysis may be performed. **For Arizona samples the surrogates must also be calibrated at a minimum of five concentrations.** Method 624 requires that the %RSD be less than 35% to use the average response factor for quantitation, the curve is to be used otherwise and should have a correlation coefficient (***r***) of $\geq$0.995. Method 8260B requires that the %RSD be less than 15% to use the average response factor for quantitation, the curve is to be used otherwise as long as ***r*** is $\geq$0.995 linear or $\geq$ 0.99 quadratic. In addition, there are calibration check compounds (CCCs) listed below which must have a %RSD less than 30% and five system performance check compounds (SPCCs) which must meet the average response factor criteria listed below. The lowest standard must be less than or equal to the reported quantitation limit and the highest standard must not exceed the linear range of the detector. Any manual integrations are documented by inclusion of the integrated signals (**before and after manual integration**) initialed, dated, and reason with the quantitation report and chromatograms. All calibration manual integrations must be approved by management. Any response factors less than 0.050 must be supported by the mass spectrum of the lowest standard. **No quadratic curves for South Carolina.**

CCCs:	1,1-Dichloroethene	Toluene
	Chloroform	Ethylbenzene
	1,2-Dichloropropane	Vinyl chloride

SPCCs:	Chloromethane	0.10
	1,1-Dichloroethane	0.10
	Bromoform	0.10
	Chlorobenzene	0.30

1,1,2,2-Tetrachloroethane	0.30

13.5.2 Initial Calibration Verification - A second source standard at the 50 µg/L (5mL purge) level is used to check the validity of the curve. The standard recovery for all analytes must be between 75 and 125%. **When analyzing samples for DOD QSM Version 4.1, DOD limits (80-120%) will be used.** If the second source recovery is above 125%, the main standard has probably deteriorated for that compound. That standard must be replaced and a new curve generated. If the second source recovery is below 75%, the second source standard has probably deteriorated for that compound and must be replaced. Any manual integrations are documented by inclusion of the integrated signals (**before and after manual integration**) initialed, dated, and reason with the quantitation report and chromatograms. All calibration manual integrations must be approved by management

13.5.3 Continuing Calibration Verification (every 12 hours) - A midpoint calibration standard (generally 50 µg/L - 5mL purge) must be analyzed and calculated against the initial calibration curve, then shown to meet the calibration check criteria before any sample analysis may be performed. Acceptance criteria for method 8260B consists of the same SPCC criteria as above and ≤20% drift or difference (calculations given in section 7.10) for the CCCs as listed above. The internal standards must also be evaluated as listed below. Any manual integrations are documented by inclusion of the integrated signals (**before and after manual integration**) initialed, dated, and reason with the quantitation report and chromatograms. All calibration manual integrations must be approved by management. Samples are then quantitated against the initial calibration curve. Note: If any compound in the continuing calibration not subject to the criteria above exceeds 30% D, it must be evaluated following the guidelines outlined in SOP QS05. If deemed acceptable, the analyst may continue analysis. **When analyzing samples for DOD QSM Version 4.1, DOD acceptance criteria of ≤ 20% drift or difference for all analytes will be used.**

NOTE: Acceptance criteria for method 624 consists of meeting recovery limits found in table 5 of the method for a QC check sample. This QC check sample is made from a separate source or lot number than the calibration standard at a concentration of 20 µg/L.

Internal standard checks

13.5.3.1 Retention time - The retention times of the internal standards in the calibration verification standard must be evaluated immediately after or during data acquisition. If the retention time for any internal standard changes by more than 30 seconds from that in the mid-point standard level of the most recent initial calibration sequence, then the chromatographic system must be inspected for malfunctions and corrections must be made, as required. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is required.

13.5.3.2 Response - If the EICP area for any of the internal standards in the calibration verification standard changes by a factor of two (-50% to +

100%) from that in the mid-point standard level of the most recent initial calibration sequence, the mass spectrometer must be inspected for malfunctions and corrections must be made, as appropriate. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is required.

14. **Procedure**

14.1 LCS - An LCS is analyzed every 12 hour tune.Using standards prepared from an alternate vendor or lot number, blank water is spiked at the 50 µg/L (5mL/soil) or 10 µg/L (25mL) level. See section 12 above for criteria and corrective action. **Note: the concentration of the LCS will be 20 µg/L when analyzing 624 samples (QC Check Sample). When analyzing samples for DOD QSM Version 4.1, DOD limits will be used.**

14.2 Method Blank - Prior to sample analysis, the system must be shown to be free of contamination through analysis of a method blank. See **Table 2** for criteria and corrective action.

14.3 Sample Analysis - Prior to analysis, the samples are prepared for chromatography using the appropriate sample preparation method (5mL water, 25mL water, low soil, high soil, etc.) See SOP 225 for preparation of a 5035 soil sample. For a 5mL/25mL water sample, use the following procedure:

14.3.1 Load the vial into the Archon autosampler in the expected position.

14.3.2 Program the Archon for the loaded vial range and necessary dilutions, making sure the programmed method is set for the same volume as the purge vessel on the front of the LSC 2000 or 3000/3100 and that the Chemstation sequence matches the Archon sequence. Note: TCLP samples are analyzed at a 10x dilution. One TCLP sample is spiked per batch at receipt of leachates.

14.3.3 After analysis of the sample has been completed, check the pH of the sample using pH paper and verify it to be less than a pH of 2 (recorded on the sequence log). If it is not, record the pH on the sequence log and generate a corrective action report. The sample report will have to be qualified for preservation if the analysis is being performed more than 7 days after sampling. [Note: TCLP samples do not require a pH check.]

14.4 **Instrument sequence**

An example of a typical instrument sequence log follows:

1-BFB Tune (12:00 am)
2-CCV
3-LCS
4-Method Blank
5-Sample
6-Sample
7-Sample
8-Sample
9-Sample
10-Sample
11-Sample
12-Sample
13-Sample
14-Sample
15-Sample
16-Sample
17-Sample MS
18-Sample MSD
19-BFB (12:00pm - 12 hours since last BFB/CCV)
20-CCV
21-LCS
22-Method Blank
23-Sample
24-Sample

14.5 Data Reduction/Evaluation - Each sample analysis sequence is documented using the computer run log generated on the chemstation. This run log is signed, dated and paginated then placed in a 3 ring binder for that instrument. After the sample has been analyzed, the data is processed through the TargetDB on Windows NT data system. Quantitative measurements are performed using the calculations found in section 15.2 of this SOP. The following must be checked to determine if the sample will need any reanalysis or dilution. Formal data evaluation is detailed in SOP QS05. **See SOP QS07 for guidance on manual integrations.**

14.5.1 Internal Standards - Areas should be within 50 to 200 percent of the area of the continuing calibration. Retention time should be within 30 seconds of the retention time of the continuing calibration. Note: criteria applies to the continuing calibration, not samples, but is used as an indication of the sample analysis validity. If not, the sample and historical data should be evaluated to determine the cause of the problem. Reanalysis is expected if it appears to be from a leak. If matrix effect is confirmed by reanalysis or historical data, complete a corrective action report and flag the affected compounds on the final report for matrix effect.

14.5.2 Surrogates – Control limits are determined by charting LCSs and method blanks. All of the surrogates must be within these limits in order for the analysis to be in control. If not, the reason for the malfunction must be determined and reanalysis may be necessary. If historical data indicates matrix, the sample would be flagged appropriately. When the surrogates exceed either the control limits, a corrective action report must be completed.

***Federal Register* Method 624 contains no criteria for surrogate recovery. When analyzing samples for DOD QSM Version 4.1, DOD limits will be used.**

Surrogate	WATER	SOIL/SEDIMENT
Dibromofluoromethane	85-120	80-125
1,2-Dichloroethane-d4	85-135	75-140
Toluene-d8	85-115	80-120
Bromofluorobenzene	80-120	80-125

14.5.3 Analyte concentration must be within the range of the calibration curve after rounding to 2 significant figures. If an analyte exceeds the curve, a dilution must be performed, the next sample must be checked for carryover and the sparge position must be checked for contamination through the analysis of a system blank. Any dilution should keep the concentration of the analyte in question within the mid-range of the curve.

14.5.4 Qualitative identification is made as indicated below.

14.5.4.1 The mass spectra are compared to reference spectra in a user-created data base especially compiled to contain relatively uncontaminated mass spectra of each target compound. Note: Such a file cannot be obtained from the daily calibrations during each 12 hour analytical period due to overlapping peaks in the mixes.

14.5.4.2 The GC/MS analyst uses intelligence guided by experience to make the identifications.In uncontaminated spectra where ions are missing due to low concentration, if the major ions are present in the correct ratios at the correct retention time, the identification will be considered positive. In contaminated spectra, special emphasis will be placed upon higher mass ions, and the major ions will usually need to be present as major components of the spectrum (either unsubtracted or subtracted) for the identification to be positive. All assessments of relative intensities of ions will be made by visual estimates from the spectra.

15. Data Analysis and Calculations

15.1 Quality Systems SOP QS09 “General and Commonly used Laboratory Calculations” provides details on general calculations used throughout the laboratory.

15.2 Calculations:

15.2.1 The RF is calculated as follows:

$$RF = \frac{A_s \times C_{is}}{A_{is} \times C_s}$$

where:

A_s = Peak area (or height) of the analyte or surrogate.

A_{is} = Peak area (or height) of the internal standard.

C_s = Concentration of the analyte or surrogate.

C_{is} = Concentration of the internal standard.

15.2.2 Calibration verification involves the calculation of the percent drift (linear) or the percent difference (average) of the instrument response between the initial calibration and each subsequent analysis of the verification standard. Use the equations below to calculate % Drift or % Difference, depending on the calibration procedure used.

$$\% \text{ Drift} = \frac{(\text{Calculated concentration} - \text{Theoretical concentration}) * 100}{\text{Theoretical Concentration}}$$

where the calculated concentration is determined from the initial calibration and the theoretical concentration is the concentration at which the standard was prepared.

$$\% \text{ Difference} = \frac{(\text{CCV RF} - \text{Average RF}) * 100}{\text{Average RF}}$$

where CCV RF is the response factor from the analysis of the verification standard and Average RF is the average response factor from the initial calibration. The % difference or % drift calculated for the calibration verification standard must be within ±20% for each CCC analyte, or for all target analytes if the CCCs are not target analytes, before any sample analyses may take place.

15.2.3 Concentration in water samples is calculated as follows: [Note: Using the units specified here for these terms will result in a concentration in units of ng/mL, which is equivalent to ug/L.]

$$\text{Concentration } (\mu g/L) = \frac{(A_s)(C_{is})(D)(V_i)}{(A_{is})(\overline{RF})(V_s)(1000)}$$

where:
A_s = Area (or height) of the peak for the analyte in the sample.
A_{is} = Area (or height) of the peak for the internal standard.
C_{is} = Concentration of the internal standard in the volume purged in ug/L.
D = Dilution factor, if the sample was diluted prior to analysis. If no dilution was made, D = 1. The dilution factor is always dimensionless.
V_i = For purge-and-trap analysis, V_i is not applicable and is set at 1.
$\overline{RF}$ = Mean response factor from the initial calibration.
V_s = Volume of the aqueous sample purged (mL). If units of liters are used for this term, multiply the results by 1000.

15.2.4 Concentration in non-aqueous samples is calculated as follows: [Note: Using the units specified here for these terms will result in a concentration in units of ng/g, which is equivalent to ug/kg.]

$$\text{Concentration } (\mu g/kg) = \frac{(A_s)(C_{is})(D)(V_i)}{(A_{is})(\overline{RF})(W_s)(1000)}$$

where: A_s, A_{is}, C_{is}, D, and $\overline{RF}$ are the same as for aqueous samples.
W_s = Weight of sample extracted (g). Either a dry weight or wet weight may be used, depending upon the specific application of the data. If units of kilograms are used for this term, multiply the results by 1000.

16. **Method Performance**

16.1 Demonstration of Capability (DOC): Each analyst must perform a DOC prior to reporting data. The analyst must prepare (for prep technicians) and analyze (analysts reviewing and reporting data) 4-LCS samples. The data is calculated for accuracy and precision requirements. The DOC form is completed by each analyst and then provided to the supervisor for further processing and approval. See **Table 2** for acceptance criteria. **When analyzing DOCs for DOD QSM Version 4.1, DOD limits will be used.**

17. **Pollution Prevention**

17.1 Quantity of chemicals purchased should be based on expected usage during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

18. **Data Assessment and Acceptance Criteria for Quality Control Measures**

18.1 Quality Control SOP QS05, "Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results", provides details on data assessment and acceptance criteria for Quality Control Measures. **Table 2** of this SOP provides information on QC samples, frequency, and the associated criteria specific to the performance of this method.

19. **Contingencies for Handling out-of-control or unacceptable data**

19.1 Quality Control SOP QS05, "Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results", provides details on handling out of control data. Table 2 within this SOP also lists corrective actions associated with the failure of the various QC samples employed for the performance of this method.

20. **Waste Management.**

20.1 Laboratory SOP QS14 on Waste Handling discusses general guidelines for the appropriate handling of wastes and the laboratory program on waste management.

21. **References**

21.1 40 CFR, Part 136; Appendix A
21.2 Test Methods for Evaluating Solid Waste, SW-846, Third Edition and updates
21.3 National Environmental Laboratory Accreditation Conference; CH. 5, 2001
21.4 USACE, EM 200-1-3; Appendix 1; Shell, 2/2001
21.5 DOD Quality Systems Manual for Environmental Laboratories version 3, 3/2005
21.6 DOD Quality Systems Manual for Environmental Laboratories version 4.1, 4/2009

22. Tables, Diagrams, Flowcharts and Validation Data

TABLE 1 – Analytes, Reporting Limit (RL), & Low Calibration Standard				
Parameter	**RL Water ug/L**	**LowCal Water ug/L**	**RL Soil ug/KG**	**LowCal Soil ug/KG**
1,1,1 Trichloroethane	1.0	1.0	5.0	2.0
1,1,1,2-Tetrachlorethane	1.0	1.0	5.0	2.0
1,1,2,2-Tetrachloroethane	1.0	1.0	5.0	2.0
1,1,2-Trichloroethane	1.0	1.0	5.0	2.0
1,1-Dichloroethane	1.0	1.0	5.0	2.0
1,1-Dichloroethene	1.0	1.0	5.0	2.0
1,2,4 Trichlorobenzene	1.0	1.0	5.0	2.0
1,2-Dibromo-3-chloropropane	1.0	1.0	5.0	2.0
1,2-Dibromoethane	1.0	1.0	5.0	2.0
1,2-Dichlorobenzene	1.0	1.0	5.0	2.0
1,2-Dichloroethane	1.0	1.0	5.0	2.0
1,2-Dichloropropane	1.0	1.0	5.0	2.0
1,3-Dichlorobenzene	1.0	1.0	5.0	2.0
1,4-Dichlorobenzene	1.0	1.0	5.0	2.0
2-Butanone	10	2.0	50	4.0
2-Hexanone	5.0	2.0	10	4.0
4-Methyl-2-pentanone	5.0	2.0	10	4.0
Acetone	10	2.0	50	4.0
Benzene	1.0	1.0	5.0	2.0
Bromochloromethane	1.0	1.0	5.0	2.0
Bromodichloromethane	1.0	1.0	5.0	2.0
Bromoform	1.0	1.0	5.0	2.0
Bromomethane	2.0	1.0	10	2.0
Carbon disulfide	1.0	1.0	5.0	2.0
Carbon tetrachloride	1.0	1.0	5.0	2.0
Chlorobenzene	1.0	1.0	5.0	2.0
Chloroethane	2.0	1.0	10	2.0
Chloroform	1.0	1.0	5.0	2.0
Chloromethane	2.0	1.0	10	2.0
Cis-1,2-Dichloroethene	1.0	1.0	5.0	2.0
Cis-1,3-Dichloropropene	1.0	1.0	5.0	2.0
Dibromochloromethane	1.0	1.0	5.0	2.0
Dibromomethane	1.0	1.0	5.0	2.0
Dichlorodifluoromethane	2.0	1.0	10	2.0
Ethylbenzene	1.0	1.0	5.0	2.0
Methylene chloride	2.0	1.0	10	2.0
M,p-Xylene	1.0	2.0	5.0	4.0
o-Xylene	1.0	1.0	5.0	2.0
Styrene	1.0	1.0	5.0	2.0

TABLE 1 – Analytes, Reporting Limit (RL), & Low Calibration Standard				
Parameter	**RL Water ug/L**	**LowCal Water ug/L**	**RL Soil ug/KG**	**LowCal Soil ug/KG**
Tetrachloroethene	1.0	1.0	5.0	2.0
Toluene	1.0	1.0	5.0	2.0
Trans-1-2 Dichlorethene	1.0	1.0	5.0	2.0
Trans-1-3-Dichloropropene	1.0	1.0	5.0	2.0
Trichloroethene	1.0	1.0	5.0	2.0
Trichlorofluroromethane	2.0	1.0	10	4.0
Vinyl chloride	2.0	1.0	10	4.0
MTBE	1.0	1.0	5.0	2.0
Naphthalene	1.0	1.0	5.0	2.0

Table 2 - Method Quality Control Requirements Summary

QC Check	Minimum Frequency / Requirements	Acceptance Criteria	Corrective Action for Failures / Data Useability
Tune	At the beginning of sequence and every 12 hours	See section 13.4 for criteria.	• Follow guidelines from SOP QS05
Calibration Curve	• Prior to analyzing any samples • A minimum of 5-points for linear fits • A minimum of 6-points for quadratic fits • Low standard at or below the RL/LOQ level	• For Linear or Quadratic calibration fits a RF of 0.995 • Average RSD for CCCs $\leq$ 30%, to use avg. RF $\leq$ 15%, Min. RF for SPCCs per method • Manual integrations on curve standards must have supervisory approval • Must follow curve processing requirements from SOP QS08	• Re-evaluate curve mix and makeup • Re-run curve • Check instrument for maintenance needs • Re-prep the curve standards Samples cannot be analyzed until there is a passing calibration
ICV	Alternate source standard to be analyzed after every calibration curve	75-125% for 8260B, 80-120% for DOD QSM 4.1	• Re-analyze an ICV from a different source • Re-prep and re-analyze the ICV • Re-calibrate and verify standard preps and sources
CCV	• At the beginning of every sequence • Every 12 hours	See section 13.5.3 for criteria.	• Follow guidelines from SOP QS05
MB	One per prep batch	• Must be < ½ the RL/LOQ	• Re-analysis to confirm the positive value • Ascertain if there are any samples within the batch that meet the MB criteria and provide the information for the decision makers • If results are between the LOD or RL/LOQ, then assess the data and notify the PM for further action • Re-prep of samples associated with the MB • NCR will be required for data reported • Final Report data flagging will be required
LCS	One per prep batch	Most stringent criteria listed within the LIMS.	• Follow guidelines from SOP QS05
LCSD	One per prep batch, when MS/MSD not included.	Criteria listed within LIMS or specified by client.	• Follow guidelines from SOP QS05

Table 2 - Method Quality Control Requirements Summary			
QC Check	**Minimum Frequency / Requirements**	**Acceptance Criteria**	**Corrective Action for Failures / Data Useability**
MS	One per prep batch, if sample volume available.	Criteria listed within LIMS or specified by client.	• Follow guidelines from SOP QS05
MSD	One per prep batch, if sample volume available.	Criteria listed within LIMS or specified by client.	• Follow guidelines from SOP QS05
Internal Standard	• A mix is used per sample post - prep	• 50 – 150 % of the IS from CCV	• If holding time is expired, fill out a NCR and follow directions from PM • Evaluate sample matrix and other applicable results to determine if re-analysis is required at a dilution • Re-injection or analysis • Re-prep followed by re-analysis • Follow guidelines from SOP QS05
Surrogates	• A mix is used per sample prior to sample prep	Criteria listed within LIMS or specified by client.	• If holding time is expired, fill out a NCR and follow directions from PM • Evaluate sample matrix and other applicable results to determine if re-analysis is required at a dilution • Re-injection or analysis • Re-prep followed by re-analysis • Follow guidelines from SOP QS05
DOC Study	• Initially per analyst prior to reporting data • Annually • Follow specific guidelines from section 16 for the preparation and analysis of DOC samples	• Must meet the criteria of the LCS for average accuracy	• Re-prep and /or re-analysis
MDL Study	Once per year	o Calculated value must be greater than 10% of the Spike Level o Calculated value must be less than the Spike level	• Re-prep and /or re-analysis • Follow guidelines from SOP QS05
LOD Verification	Every quarter	o Parameter must be detected o Check for Ion Abundance on MS methods o the response must be 3-times the noise level	• Re-prep and /or re-analysis • Follow guidelines from SOP QS05

Table 2 - Method Quality Control Requirements Summary			
QC Check	**Minimum Frequency / Requirements**	**Acceptance Criteria**	**Corrective Action for Failures / Data Useability**
LOQ Verification	Every quarter	○ Bias Requirement: Inorganics 50-150% Organics 10-150% ○ The LOQ value must be greater than the LOD value	• Re-prep and /or re-analysis • Follow guidelines from SOP QS05

Table 3, Technical Completeness / Accuracy Checklist

1. Were all the QC check elements analyzed – refer to Table 2 of the SOP
2. Were the QC criteria met
3. In cases of failures, was there an NCR written
4. Were all manual integrations signed
5. Were dilution factors applied correctly
6. Was there supervisory approval for manual integrations on standards and QC samples
7. Was the data uploaded into LIMS via direct upload – if yes, then was a cross check subset of the uploaded values performed
8. If the data was entered into LIMS manually, was a check of all entered values performed
9. Was the red marked data in LIMS checked for accuracy and the corresponding hard copy data documented appropriately
10. Were proper data qualifiers applied to the data in LIMS
11. Was the hard copy package checked for completeness to include all data for the sequence such that the data reviewer could reconstruct sample analyses and validate / approve the data

Table 4, Data Reviewers Checklist (Prior to approving data)

1. Does the hard copy raw data (or electronic raw data) package look complete and include all data points
2. Were QA objectives met and for failures were the appropriate actions taken
3. For direct uploads to LIMS, did a subset cross check match the raw data
4. Did all the manual entries into LIMS match the raw data
5. Were there appropriate signatures and documentation on the raw data
6. Were appropriate LIMS flags used
7. Were manual integrations signed
8. Were manual integrations for calibration and QC samples approved by supervisor
9. Were manual calculations verified

EMPIRICAL LABORATORIES, LLC
STANDARD OPERATING PROCEDURE

ORGANICS: SOP 218 **REVISION #: 06** **EFFECTIVE DATE: 093009**

1, 2-DIBROMOETHANE AND 1, 2-DIBROMO-3-CHLOROPROPANE BY GC/ECD EPA METHODS 504.1 & SW-846 8011

APPROVALS:

Lab Director: ______________ Date: 10/5/09

Data Quality Manager: ______________ Date: 10/5/09

Section Supervisor: ______________ Date: 10/7/09

Changes Summary

Revision 06, 9/30/09

- The SOP is an update from Revision 05 dated 08/28/08
- The SOP is formatted to include all 22-elements required per the NELAC standards
- The laboratory's revision of all technical SOPs now includes a Table of Contents that provides the map of the technical information contained within the SOP.
- Additional requirements, based upon the DoD QSM 4.1, have been integrated into the routine sample flow; however, if the requirement is different from routine sample flow, then the requirement is outlined and documented as such to be followed only when DoD samples are analyzed.

Table of Contents

1.0 Identification of the Test Method

1.1 This SOP is compliant with EPA method 504.1 and SW-846 method 8011.

2.0 Applicable Matrix or Matrices

2.1 This SOP is applicable to the determination of 1,2-dibromoethane (EDB) and 1,2-dibromo-3-chloropropane (DBCP) in drinking water and groundwater.

3.0 Detection Limit

3.1 The method has been shown to be useful over a range of 0.03 µg/L to 200 ug/L for EDB and DBCP for groundwater. The MDL has been experimentally determined to be 0.01 µg/L.

4.0 Scope of Application, Including Components to Be Analyzed

4.1 Each parameter that is analyzed and reported under the scope of this SOP is listed in **Table 1** of this SOP. This table also lists the associated Reporting Limit (also defined as the LOD) and the lowest Calibration level for each analyte. When applicable, surrogate and Internal Standard Analytes are listed and indicated as such within this table.

4.2 The GC/ECD system is used to analyze 1,2-dibromoethane (EDB) and 1,2-dibromo-3-chloropropane (DBCP) in ground water. This SOP will describe the analysis of these compounds using a temperature programmable gas chromatograph configured with dual electron capture detectors (ECD) and a capillary column splitless injector.

4.3 Extreme care should be taken when working with pure standard and stock standard solutions of these compounds and all handling of standards should be done in a hood. These compounds have been classified as known or suspected human or mammalian carcinogens.

5.0 Summary of the Test Method

5.1 Thirty-five mL of sample are extracted with 2 mL of hexane. One µL of the extract is then injected into the gas chromatograph equipped with dual linearized electron capture detectors for separation and analysis. All standards, QC spikes, and blanks are prepared, extracted, and analyzed using this same method.

6.0 Definitions

6.1 Laboratory Quality System SOP QS08 "Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures" provides information on the commonly used definitions.

7.0 Interferences

7.1 Interferences can come from the extracting solvent or reagents. Reagent Blanks must be analyzed to check for interferences with the analysis.

7.2 Interferences can come from other organic compounds contained in the sample.

7.3 Dibromochloromethane is a common chlorinated drinking water contaminant, which in high concentrations may mask low concentrations of EDB. Therefore, special care should be taken in the identification and confirmation of EDB.

8.0 Safety

8.1 Laboratory SOP QS13 “Safety Program & Chemical Hygiene Plan” discusses the safety program that is to be followed labwide.

9.0 Equipment & Supplies

9.1 Syringes – 10ul, 25ul, 100ul, and 5ml (Hamilton 702N or equivalent)
9.2 Vials – 2ml amber autosampler with Teflon-lined screw cap; 40ml VOA with Teflon-lined screw cap; 12ml with Teflon-lined screw cap
9.3 Transfer Pipets
9.4 Graduated Cylinder (Glass) – 50ml
9.5 Volumetric Flask – 10ml
9.6 Balance – top loading, 0.01g
9.7 Gas Chromatograph – Agilent 6890 (temperature programmable)
9.8 Autosampler – HP-7683 injector
9.9 Columns
  9.9.1 Column A – Phenomenex ZB-Multiresidue-1, 30m, 0.32mm ID, 0.50um thickness or equivalent.
  9.9.2 Column B (confirmation column) – Phenomenex ZB-Multiresidue-2, 30m, 0.32mm ID, 0.25um thickness or equivalent.
9.10 Data System
  9.10.1 Acquisition Software – HP Chemstation system is interfaced to the GC. The system acquires and stores data throughout the chromatographic program.
  9.10.2 Data Processing Software – TARGET Chemserver 4920 data system is interfaced to the HP Chemstation. The system accepts, processes, and stores acquired data.

10.0 Reagents and Standards

10.1 The laboratory’s LIMS system allows for complete documentation and for the traceability of reagents and standards used within the laboratory. The following information relates to the specific reagents and standards used for the performance of the method:

Reagent grade chemicals shall be used in all tests. All reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination. Certified stock standards are purchased from Restek, Protocol, Ultra and other vendors depending on their availability. The date they are received is noted on the label or container they are received in and in the LIMS system. The date the standards are opened they are recorded and given a sequential number in the LIMS system. All stock standards are stored at 4 ° C.

10.2 Organic-free reagent water from our laboratory modulab analytical purification system.
10.3 Hexane - Pesticide grade
10.4 Methanol – Purge and Trap.
10.5 Sodium Chloride - demonstrated to be interference free. If needed, sodium chloride can be placed in the muffle furnace at room temperature and increase to 400° for 30min. Place in a bottle and cap.

10.6 Intermediate stock standards are prepared from these certified stock standards. All intermediate standards (in methanol) are prepared at a concentration that can be easily diluted (see below) to prepare aqueous calibration standards that will bracket the working calibration range. The information concerning the preparation of these standards is noted in the LIMS system, detailing how they were made, solvent (methanol, laboratory reagent blank water) used, date made, expiration date and given a unique sequential number Standards may be used for at least four weeks. A second source check is required every time a new standard or standard curve is implemented. The intermediate stock standard is then stored at 4 C.

10.7 Calibration standards must be prepared at a minimum of five concentration levels for each analyte through dilution of the intermediate stock standard into laboratory reagent blank water. One of the concentration levels should be near, but above, the method detection limit (MDL). The remaining concentration levels should correspond to the expected range of concentrations found in real samples or define the working range of the GC-ECD system. Typical concentration levels are 0.03, 0.05, 0.10 ,0.15, and 0.20 µg/L In order to prepare accurate aqueous standard solutions, the following precautions must be observed:

10.7.1 Use a 25 µL Hamilton 702N microsyringe or equivalent (variations in needle geometry will adversely affect the ability to deliver reproducible volumes of methanolic standards into water).

10.7.2 Rapidly inject the alcoholic standard into the filled volumetric flask or vial. Remove the needle as fast as possible after injection.

10.7.3 Mix the aqueous standards by inverting the flask or vial three times only.

11.0 Sample Collection, Preservation, Shipment, and Storage

11.1 Quality Systems SOP QS10 related to Sample Receipt, Handling, & Processing provides details for collection, preservation, shipment, and storage.

11.2 Samples are collected in 40ml vials and stored at 4ºC.

11.3 Holding time is 14 days from sample collection to extraction and analysis.

12.0 Quality Control

12.1 Quality Systems SOP QS08 “Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures” outlines details related to laboratory wide protocols on quality control.

12.2 An initial demonstration must be performed by each analyst performing this method. Four LCSs are analyzed at 0.10ug/L. See **Table 2** for acceptance criteria.

12.3 For method 504.1, each day a low level standard at 0.02ug/L should be analyzed to check for adequate sensitivity.

12.4 A CCV (QC check standard) at a concentration of 0.10 µg /L with both analytes is required each batch or at a 5% frequency whichever comes first. The recovery must be between 60% and 140%. **When analyzing samples for DOD QSM Version 4.1, DOD acceptance criteria of $\leq$ 20% drift or difference for all analytes will be used.** If there is failure to meet this criteria, check the analytical system to locate and correct any problem and/or a repeat with another check standard before samples can be analyzed.

12.5 An LCS (QC reference sample) is required each batch at a concentration of 0.10 µg/L to check the calibration standard. The recovery must be between 60% and 140% before the curve can be used to analyze samples. If this criterion is not met, recheck the analytical system for errors and repeat the test. **When analyzing samples for DOD QSM Version 4.1, DOD acceptance criteria of ≤ 20% drift or difference for all analytes will be used.**

12.6 Check the performance of the analytical system daily by using data gathered from analyses of blanks, standards, and replicate samples. System maintenance can be indicated through the performance of the analyses.

12.6.1 Peak tailing problems which show up in the chromatography are traceable to active sites on the GC column or to the detector.

12.6.2 Check precision between replicate analyses. The system should perform with an average relative standard deviation of less than 10%.

Poor precision could indicate pneumatic leaks, especially at the injection port.

12.6.3 Maintenance of the GC system should be performed and logged into the maintenance logbook. Maintenance should include the following:

12.6.3.1 Clean or deactivate glass injection port insert or replace with a cleaned and deactivated insert.

12.6.3.2 Break off the first few inches of the injection port side of the column.

12.6.3.3 Remove the column and column back-flush according to the manufacture's specifications.

12.6.3.4 If all else fails to correct the problem, the metal injection port body may need to be deactivated or the column replaced.

12.7 RT Windows - Retention time criteria set forth in SW-846 method 8000B section 7.6 are used to set retention time windows. New in-house retention time windows are established after every major change to the system (new column or temperature program). If the established retention time window is less than +/-0.03 minutes, the window defaults to +/-0.03 minutes. Retention times are updated with the first CCV of the day or the mid-level standard of the curve if samples are analyzed directly after a curve.

12.8 Sample analyses will begin after the analyst performs the various checks just mentioned. If there was a problem in meeting the QC criteria then system maintenance may be required (see above) as stated.

13.0 Calibration and Standardization

13.1 Quality Systems **SOP QS08 "**Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures" related to Calibration Procedures provides laboratory wide protocols for calibration and standardization.

13.2 An extracted five-point calibration curve must be introduced and analyzed for each analyte using the appropriate instrument parameters. One level of concentration containing EDB and DBCP should be near but above the MDL for each compound (reference SW-846 Method 8011 Section 7.2).

13.2.1 Tabulate peak height or area response versus the concentration in the standard and prepare a calibration curve for each compound. Alternatively, if the ratio of response to concentration (calibration factor) is a constant over the working range (< 10% relative standard deviation 8011 and < 20% for 504.1), linearity can be assumed and the average calibration factor can be used in place of a calibration curve (linear corr. ≥ 0.995). The curve is verified using a second source check. All calibration integrations must be verified and any manual integrations are documented by the inclusion of the chromatogram (which includes the before and after chromatograms of the peak integrations) with the quantitation report. See **SOP QS07** for guidance.

13.2.2 An Initial Calibration Verification (ICV) standard is prepared in the same manner as the QC reference sample and analyzed after the initial calibration but before client samples. The ICV must pass acceptance criteria (see **Table 2**) before the analysis of samples can continue.

14.0 Procedure

14.1 Samples and standards are removed from storage and left to equilibrate to room temperature. Sample and/or standard extraction must be performed in an organic solvent free environment.

14.2 For samples and field blanks contained in a 40 mL VOA vial remove the vial cap and measure off and discard 5 mL of sample using a 5 mL transfer pipette. Replace the vial cap and weigh and record the weight of the container with contents to the nearest 0.1 g. This weight will be used for subsequent sample volume determination (see below).

14.3 For calibration standards, check standards, QC reference samples, and blanks, measure a 35 mL volume (laboratory reagent blank containing the appropriate spike) using a 50 mL graduated glass cylinder and transfer to a 40 mL vial.

14.4 Begin the extraction of the samples by removing the cap again and adding 7 g of NaCl to all samples. Recap the sample container and dissolve the NaCl by shaking by hand for about 20 seconds

14.5 Remove the cap again and using a 1mL syringe, add 2.0 mL of hexane. Recap and shake vigorously by hand for 1.0 minute. Allow the water and hexane to separate (if stored at this stage, keep the container upside down).

14.6 Remove the cap and carefully transfer a sufficient amount (0.5 - 1.0 mL) of the hexane layer into a vial using a disposable glass pipette. Repeat this step to a second vial being careful not to include any of the water phase. Reserve this second vial at 4 ° C for reanalysis if necessary.

14.7 Determination of the sample and field blank volume is done by totally removing any sample/hexane mixture left in the vial (this is discarded). Re-weigh the empty vial including the cap and calculate the net weight of sample by the difference to the nearest 0.1 g from the previous weight determination.

15.0 Data Analysis and Calculations

15.1 Quality Systems SOP QS09 “General and Commonly used Laboratory Calculations” provides details on general calculations used throughout the laboratory.

15.2 GC oven conditions

15.2.1 Injector temperature: 200°C.

15.2.2 Detector temperature: 290°C.

15.2.3 Carrier gas (Helium) 1.9 ml/min. constant flow

15.2.4 Temperature program:

Initial temperature: 35°C, hold for 2.0 min.

Program: Ramp at 12°C / min. to 290°C

15.3 Transfer all samples (including standards, blanks, and unknown samples) to an autosampler set up for a 1.0 µL injection. Analyze according to previously stated instrument parameters (see above).

15.4 Analytes are identified by retention time on a primary column with confirmation by retention time on a secondary or confirmation column. Which column is used for primary/confirmation is determined by the chromatography in the region of the compound. If both columns are equivalent, the higher concentration is reported.

15.5 Samples are reduced using the TARGET Chemserver data system. The analytes detected in the samples are calculated using the calibration factor or calibration curve giving an uncorrected concentration of each analyte in the sample (reference SW-846, Method 8011, Section 7.7). These results are then referenced to any dilutions and the initial sample volume to complete the quantitation of analyte in the sample. The final analytical results are reported in µg / L.

15.6 Any questions left unanswered by this SOP should be clarified by reading the referenced method. If questions still remain unanswered, check with the Section Manager, and/or Technical Director or Quality Assurance Manager.

16.0 Method Performance

16.1 Demonstration of Capability (DOC): Each analyst must perform a DOC prior to reporting data. The analyst must prepare (for prep technicians) and analyze (analysts reviewing and reporting data) 4-LCS samples. The data is calculated for accuracy and precision requirements. The DOC form, as listed within section 2.5 of the Quality is completed by each analyst and then provided to the supervisor for further processing and approval. See **Table 2** for acceptance criteria. **When analyzing DOCs for DOD QSM Version 4.1, DOD limits will be used.**

See method 8011 and 504.1 for method performance.

17.0 Pollution Prevention

17.1 Quantity of chemicals purchased should be based on expected usage during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

18.0 Data Assessment and Acceptance Criteria for Quality Control Measures

18.1 Quality Control SOP QS05, "Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results", provides details on data assessment and acceptance criteria for Quality Control Measures. **Table 2** of this SOP provides information on QC samples, frequency, and the associated criteria specific to the performance of this method.

19.0 Contingencies for Handling out-of-control or unacceptable data

19.1 Quality Control SOP QS05, "Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results", provides details on handling out of control data. **Table 2** within this SOP also lists corrective actions associated with the failure of the various QC samples employed for the performance of this method.

20.0 Waste Management

20.1 Laboratory SOP QS14 on Waste Handling discusses general guidelines for the appropriate handling of wastes and the laboratory program on waste management.

21.0 References

21.1 *Test Methods for Evaluating Solid Waste Physical/Chemical Methods, SW-846; Third Edition (Update III);* Method 8011.

21.2 *Method 504.1, 1,2-Dibromoethane (EDB) and 1,2-Dibromo-3-chloropropane (DBCP), and 1,2,3-Trichloropropane in Water by Microextraction and Gas Chromatography,* USEPA, 1995.

22.0 Tables, Diagrams, Flowcharts and Validation Data

22.1 Table 1, all applicable parameters, including the surrogates and internals with the applicable RL and lowest calibration standard.

22.2 Table 2, for all technical methods, should always be the QA/QC summary table and I am including a format for this at the end.

22.3 Table 3, Technical Completeness / Accuracy Checklist

22.4 Table 4, Data Reviewers Checklist

22.5 Validation data would be actual documentation (eg: a pdf email from a regulator explaining the approach to a method, etc.) or a side by side study performed to reach to our approach on how we handle the method.

TABLE 1 – Analytes, Reporting Limit (RL), & Low Calibration Standard (Units ug/L)		
Parameter	**RL**	**LowCal**
EDB (1,2-dibromoethane)	0.03	0.03
DBCP (1,2-dibromo-3-chloropropane)	0.03	0.03

Table 2 - Method Quality Control Requirements Summary			
QC Check	**Minimum Frequency / Requirements**	**Acceptance Criteria**	**Corrective Action for Failures / Data Useability**
Calibration Curve	• Prior to analyzing any samples • A minimum of 5-points for linear fits • A minimum of 6-points for quadratic fits • Low standard at the RL/LOD level	• For Linear or Quadratic calibration fits a RF of 0.995 • Average RSD $\leq$ 20% (504.1) • Average RSD $\leq$ 10% (8011) • Manual integrations on curve standards must have supervisory approval • Must follow curve processing requirements from SOP QS08	• Re-evaluate curve mix and makeup • Re-run curve • Check instrument for maintenance needs • Re-prep the curve standards Samples cannot be analyzed until there is a passing calibration
ICV	Alternate source standard to be analyzed after every calibration curve	Must meet the criteria of the CCV	• Re-analyze an ICV from a different source • Re-prep and re-analyze the ICV • Re-calibrate and verify standard preps and sources
CCV	• At the beginning of every sequence • For every 10-client samples • The concentration should be varied from low to mid range	• $\leq$ 20% drift or difference for all analytes	• Follow guidelines for SOP QS05
Closing CCV	• At the end of every sequence	• $\leq$ 20% drift or difference for all analytes	• Follow guidelines for SOP QS05
MB	One per prep batch	• Must be less than the LOD	• Re-analysis to confirm the positive value • Ascertain if there are any samples within the batch that meet the MB criteria and provide the information for the decision makers • If results are between the LOD or RL/LOQ, then assess the data and notify the PM for further action • Re-prep of samples associated with the MB • NCR will be required for data reported • Final Report data flagging will be required
LCS	One per prep batch	Most stringent criteria listed within the LIMS.	• Follow guidelines from SOP QS05

Table 2 - Method Quality Control Requirements Summary			
QC Check	**Minimum Frequency / Requirements**	**Acceptance Criteria**	**Corrective Action for Failures / Data Useability**
LCSD	One per prep batch, when MS/MSD not included.	Criteria listed within LIMS or specified by client.	• Follow guidelines from SOP QS05
MS	One per prep batch, if sample volume available.	Criteria listed within LIMS or specified by client.	• Follow guidelines from SOP QS05
MSD	One per prep batch, if sample volume available.	Criteria listed within LIMS or specified by client.	• Follow guidelines from SOP QS05
Surrogates	• A mix is used per sample prior to sample prep	Criteria listed within LIMS or specified by client.	• If holding time is expired, fill out a NCR and follow directions from PM • Evaluate sample matrix and other applicable results to determine if re-analysis is required at a dilution • Re-injection or analysis • Re-prep followed by re-analysis • Follow guidelines from SOP QS05
DOC Study	• Initially per analyst prior to reporting data • Annually • Follow specific guidelines from section 16 for the preparation and analysis of DOC samples	• Average percent recovery should be between 70-130%, with a 20% standard deviation.	• Re-prep and / or re-analysis
MDL Study	Once per year	• Calculated value must be greater than 10% of the Spike Level • Calculated value must be less than the Spike level • Percent recovery must be between 60-140% of the true value of each analyte.	• Re-prep and / or re-analysis • Follow guidelines from SOP QS05
LOD Verification	Every quarter	• Parameter must be detected • 2nd column / detector confirmation is required • the response must be 3-times the noise level	• Re-prep and / or re-analysis • Follow guidelines from SOP QS05

Table 2 - Method Quality Control Requirements Summary			
QC Check	**Minimum Frequency / Requirements**	**Acceptance Criteria**	**Corrective Action for Failures / Data Useability**
LOQ Verification	Every quarter	• Bias Requirement: Organics 10-150% • The LOQ value must be greater than the LOD value	• Re-prep and / or re-analysis • Follow guidelines from SOP QS05

Table 3, Technical Completeness / Accuracy Checklist

1. Were all the QC check elements analyzed – refer to Table 2 of the SOP
2. Were the QC criteria met
3. In cases of failures, was there an NCR written
4. Were all manual integrations signed
5. Were dilution factors applied correctly
6. Was there supervisory approval for manual integrations on standards and QC samples
7. Was the data uploaded into LIMS via direct upload – if yes, then was a cross check subset of the uploaded values performed
8. If the data was entered into LIMS manually, was a check of all entered values performed
9. Was the red marked data in LIMS checked for accuracy and the corresponding hard copy data documented appropriately
10. Were proper data qualifiers applied to the data in LIMS
11. Was the hard copy package checked for completeness to include all data for the sequence such that the data reviewer could reconstruct sample analyses and validate / approve the data

Table 4, Data Reviewers Checklist (Prior to approving data)

1. Does the hard copy raw data (or electronic raw data) package look complete and include all data points
2. Were QA objectives met and for failures were the appropriate actions taken
3. For direct uploads to LIMS, did a subset cross check match the raw data
4. Did all the manual entries into LIMS match the raw data
5. Were there appropriate signatures and documentation on the raw data
6. Were appropriate LIMS flags used
7. Were manual integrations signed
8. Were manual integrations for calibration and QC samples approved by supervisor
9. Were manual calculations verified

GC/MS VOLATILE NON-AQUEOUS MATRIX EXTRACTION USING SW-846 METHOD 5035 FOR 8260B ANALYSIS

SOP NUMBER: **SOP-225**

REVISION NUMBER: **8**

APPROVED BY:

SECTION MANAGER

QUALITY ASSURANCE MANAGER

EFFECTIVE DATE: **09/24/08**

DATE OF LAST REVIEW: **09/24/08**

GC/MS - VOLATILE
NON - AQUEOUS MATRIX EXTRACTION
USING SW-846 METHOD 5035

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to detail soil sample preparation for EPA method SW-846 5035. Soil samples should be sampled in the field using the EnCore™ sampler then shipped to the lab within 24 hours for preservation, storage and analysis. This SOP should be used in conjunction SOP-202, which details the analytical technique.

2.0 SUMMARY

Samples are collected in EnCores or Terracore vials. EnCore samples have to be prepped within 48 hrs of collection. Terracores are shipped already prepared.

3.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

EnCores are prepped within 48 hrs of collection in sodium bisulfate and refrigerated at 4°C or in reagent water and frozen. Terracores are prepped in the field in sodium bisulfate or water. Sodium bisulfate Terracores are refrigerated at 4°C and those prepped in reagent water are frozen. Holding Time is 14 days

4.0 INTERFERENCES AND POTENTIAL PROBLEMS

Sample vials can be a source of contamination. Vials should be checked for contamination before use. Samples can be contaminated during sample prep. Prep blanks should be prepared at the same time as the samples to check for contamination.

5.0 EQUIPMENT AND MATERIALS

- Sample Containers – 40mL VOA vials with low bleed septa. Available from ESS (Part No. PC0040-0300 pack of 72), alternate sources are possible but must be checked for contaminants before use. ESS also supplies pre-prepped vials with the preservative and stirbar (Part No. PC4039-5035 pack of 72).
- Varian Archon 51 position programmable autosampler, or equiv.
- Top-loading balance – capable of accurately weighing to 0.01g.

- 1-10 mL Adjustable Dispenser, Model 400 Series, Oxford pipettor. Available from Oxford (Part No. 8885-040009).
- Spatula, stainless steel – narrow enough to fit into a sample vial.
- Magnetic stirring bars – PTFE- or glass-coated, of the appropriate size to fit the sample vials. Available from A. Daigger (Part No. WX22782A, case of 50).
- EnCore™ sampler – (En Chem, Inc., 1795 Industrial Drive, Green Bay, WI 54302), or equivalent. Necessary for field sampling crew.
- Terracore Vials- Available from QEC.
- Balance weights – used to calibrate the balance.
- Labels.

6.0 REAGENTS

- Reagent Water - Reagent water is NANO PURE WATER from source in the instrument lab, which is then purged with helium before use.
- Methanol, CH_3OH – purge-and-trap quality, or equivalent. Store away from other solvents.
- Sodium bisulfate, $NaHSO_4$ – ACS reagent grade, or equivalent. Available from Aldrich (Part No. 30,782-3).
- Sodium bisulfate solution – Prepare by adding 200 grams of $NaHSO_4$ (ACS reagent grade, or equivalent) to 1000 milliliters of helium-purged reagent water. Record the vendor and lot number of the $NaHSO_4$ in the Standards and Reagents Logbook. Each standard/reagent that is prepared is recorded in the logbook and given a sequential number. The label is completed with the standard/reagent number, name, preparation date, expiration date, solvent and analyst initials. The solution should be discarded after six months or sooner if it shows signs of contamination.

7.0 SAMPLE COLLECTION

As with any sampling procedure for volatiles, care must be taken to minimize the disturbance of the sample in order to minimize the loss of volatile compounds. Always wear gloves whenever handling the tared sample vials. Several techniques may be used to perform the transfer of the sample to the relatively narrow opening of the low concentration soil vial such as the EnCore™ sampler, a cut off disposable plastic syringe, or a stainless steel spatula. We prefer to use the EnCore™ sampler.

7.1 The EnCore™ sampler is both a sampler and a container for low-level and high level soils. It is designed to collect an average weight with the exact weight to be determined in the lab. It is disposable and is also designed to have zero headspace. The EnCore™ sampler will require the field personnel to get the sample to the laboratory within 24-36 hours of collection. The laboratory needs to be contacted prior to sample collection to ensure that all necessary containers (with or without preservative) are available and that the proper sampling technique is used.

7.2 All low-level soil samples must be collected in duplicate to allow the laboratory an additional sample for reanalysis. A third sample should be collected for preparation of a high-level sample. This sample would be prepared at the same time as the "low-level" sample. (Some projects may not require the "low-level" detection limits, in this case only the high level sample preparation would be required.) A fourth sample may be collected to enable the laboratory to perform a pretest on the soil to determine if the soil sample contains carbonate minerals that will effervesce upon contact with the acidic sodium bisulfate preservative solution in the low concentration sample vial. The additional soil samples must be collected from the same soil stratum or the same section of solid waste being sampled and within close proximity to the location from which the original sample was collected. Additional bulk samples should be collected for screening and dry weight determination without the preservative. Note: If the low-level sample cannot be preserved with sodium bisulfate, the remaining low-level sample aliquot(s) is(are) transferred to a pre-weighed vial containing 5 mL of reagent water. The sample in the unpreserved vial must either be analyzed immediately (within 48 hours of collection) or frozen within the 48 hour time frame and then analyzed within the 14 day holding time.

8.0 PROCEDURE

8.1 Log-in personnel will log the samples in, place them in the Soil walk-in cooler assigned for volatile sample storage and notify the Organic Lab Manager that samples are in-house for 5035 preparation.

8.2 The Organic Lab Manager or designee will determine the amount of time remaining on the 48 hour EnCore™ holding time and assign the task of preserving the samples.

8.3 Samples received from the field should be designated for low-level, high-level or % solids/screening (this fraction should be in a regular soil jar, if it is not, it will require transfer to a VOA vial). Each low-level and high-level sample must be preserved appropriately as follows:

8.3.1 Organize the VOA vials required and label them with the sample number, date and LOW or HIGH for either low-level or high-level preservation. The LOW level VOA vials should have gray caps and septa if using the ESS brand.

8.3.2 Get the samples from the Hobart assigned for volatile sample storage and log them out.

8.3.3 Enter the sample numbers in the soil sample preparation logbook and add a sample preparation/storage blank to the book for each level being prepared (HIGH/LOW). There must be a line in the logbook for each sample vial being prepared (i.e. if there are 2 low-level samples and 1 high-level sample, the sample number should be listed in the logbook 3 times- use a,b,c to designate each vial associated with the same sample).

8.3.4 Using an adjustable Oxford pipettor, add 5 mL P&T methanol to each of the vials marked HIGH. Then record the vendor & lot number of methanol and the exact volume of methanol added to each sample in the sample preparation logbook. If the vial is not to be used immediately, weigh the vial to the nearest 0.01g and record the weight on the vial. The vial weight must be verified to be within ±0.01g of this value before using for sample preparation.

8.3.5 For each of the vials marked LOW, add 5 mL of sodium bisulfate or reagent water if frozen and record the reagent number in the sample preparation logbook. Add a magnetic stir bar to each vial. If pre-prepped vials from ESS (or equivalent) are used, this step is unnecessary but the lot number and the pre-prepped status must be recorded in the preparation log.

NOTE: Soil samples that contain carbonate minerals (either from natural sources or applied as an amendment) may effervesce upon contact with the acidic preservative solution in the low concentration sample vial. If the amount of gas generated is very small (i.e., several mL), any loss of volatiles as a result of such effervescence may be minimal if the vial is sealed quickly. However, if larger amounts of gas are generated, not only may the sample lose a significant amount of analyte, but the gas pressure may shatter the vial if the sample vial is sealed. Therefore, when samples are known or suspected to contain high levels of carbonates, a test sample should be collected, added to a vial, and checked for effervescence. If a rapid or vigorous reaction occurs, discard the sample and place low concentration samples in vials that contain 5ml water and a stir bar. This sample must be frozen in a slanted position until analysis or analyzed within 48 hours of sampling. Notify the Organic Lab Manager if this occurs, note this in the sample preparation logbook and generate a CAR to document the problem.

8.3.6 Place the vial (LOW/HIGH) on the top-loading balance, tare the vial then extrude the sample into the vial and record the weight of the sample in the sample preparation logbook. Make sure the lip of the vial does not have any soil on it, which might cause a leak, cap the vial tightly and mark the weight on the sample label.

8.3.7 Place the preserved samples in a box, return them to the Hobart assigned for volatile sample storage and log them back in.

9.0 ANALYSIS

9.1 Samples are analyzed by USEPA SW-846 methods 5035/8260B (low-level) using the Archon 51 position autosampler in conjunction with the GC/MS or 5030B/8260B (high-level) using any purge and trap instrument in conjunction with the GC/MS. For method 5035, the prepared low-level vials are placed in the Archon autosampler. The autosampler is programmed to add the appropriate internals and surrogates to each sample. Use of the autosampler is covered in the owners manual. Calibration of the analytical instrument with subsequent analysis of the samples is covered under SOP-202.

9.2 Determination of % Dry Weight – Weigh 5-10 grams of the sample from the bulk jar used for dry weight analysis in a tared crucible or aluminum pan. Dry overnight at 105°C. Allow to cool in a dessicator before weighing. Calculate % dry weight as follows:

$$\% \text{ dry weight} = \frac{\text{g of dry sample}}{\text{g of sample}} \times 100$$

10. HEALTH,SAFETY,WASTE MANAGEMENTAND POLLUTION PREVENTION

10.1 Care should be used in handling all samples. Safety glasses must be worn in the lab at all times. The use of blue nitrile gloves and lab coats is highly recommended.

10.2 Research into expected sample content and concentration should be done in order to be prepared for additional safety considerations. Generally, any samples that need special consideration have applicable notes on the sample logs.

10.3 MSDS sheets are available for all reagents and standards that have been purchased. These are located in the bookshelves across from the Quality Assurance Officers cube.

10.4 Please see Waste Disposal, SOP-210 and SOP-405 for proper disposal of waste coming from this area within our laboratory. Quantity of chemicals purchased should be based on expected usage during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

REFERENCES

1.*Test Methods for Evaluating Solid Waste Physical/Chemical Methods, SW-846; Third Edition (Update III)*; Method 5035.

DEFINITIONS

Refer to SOP-431 for common environmental laboratory definitions.

EMPIRICAL LABORATORIES, LLC
STANDARD OPERATING PROCEDURE

ORGANICS: SOP 338 **REVISION #: 08** **EFFECTIVE DATE: 042910**

FLPRO
METHOD FOR DETERMINATION OF PETROLEUM RANGE ORGANICS

APPROVALS:

Lab Director: D. [signature] Date: 4/29/10

Data Quality Manager: [signature] Date: 4/29/10

Section Supervisor: [signature] Date: 4/29/10

Changes Summary

Revision Date: 042910

- The SOP is a revision of rev07 dated 022410.
- Additional requirements, based upon the DoD QSM 4.1, have been integrated into the routine sample flow; however, if the requirement is different from routine sample flow, then the requirement is outlined and documented as such to be followed only when DoD samples are analyzed.
- Table 2 has been updated to reflect method surrogate limits and in-house action limits for samples.

Table of Contents

PETROLEUM HYDROCARBONS

1. **Test Method**

   1.1. This SOP is based upon method FL PRO.

2. **Applicable Matrix**

   2.1. This SOP is applicable to the determination of the concentration of Petroleum Hydrocarbons in ground water, sediments, and wastes in the alkane range of C-8 to C-40.

3. **Detection Limit**

   3.1. The detection limit for method FL-PRO is 0.085mg/L in water and 5.6 mg/Kg in soil.

4. **Scope and Application**

   4.1. Water samples are preserved with sulfuric acid to pH <2 and cooled to 4°C. Soils are stored at 4°C. Waters must be extracted within 7 days and soils within 14 days from collection and analyzed within 40 days of extraction. Extracts are kept at 4°C. Observe all safety guidelines when handling samples and extracts.

   4.2. This method is recommended for use by experienced analysts or under the close supervision for such qualified personnel.

5. **Summary of Record**

   5.1. Samples are extracted via proper extraction methods. A 1µL aliquot of the extract is injected into a GC system equipped with a flame ionization detector (FID). Quantification is based on the detector response in comparison to a series of alkane standards.

6. **Definitions**

   6.1. Laboratory Quality System SOP QS08 "Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures" provides information on the commonly used definitions.
   6.2. Petroleum Hydrocarbons: All chromatographic peaks, both resolved and unresolved, eluting between the peak of n-octane (n-C8) and the peak end after n-tetracontane (n-C40). Quantitation is based on direct comparison of the area within this range to the total area of the Petroleum Hydrocarbon standard as determined from the FID response using baseline – baseline integration.
   6.3. Petroluem Hydrocarbon Standard: A 17-component mix of all even-numbered alkanes from C8 to C40. This standard serves as a quantitation standard and a retention time window defining Petroleum Hydrocarbons.

7. **Interferences**

   7.1. All materials utilized during this analysis and the GC system must be demonstrated to be free from interference. Running frequent instrument blanks and methods blanks along with using pure, GC grade solvents will assist with the monitoring of interference's within the analytical system.
   7.2. Any interference's co-extracted with the samples will vary considerably from source to source. Individual samples may require additional cleanup.

8. Health and Safety

8.1. Laboratory SOP QS13 “Safety Program & Chemical Hygiene Plan” discusses the safety program that is to be followed lab wide.

9. Equipment and Supplies

9.1 Separatory Funnel - 2-Liter with Teflon stopcock
9.2 Beakers- 250 ml
9.3 Turbo-Vap evaporation tube - 200 mL tube made by Zymark to fit into Turbo-Vap evaporator
9.4 Metal or wood rack - capable of holding at a minimum six glass evaporation tubes
9.5 Turbo-Vap Evaporator - heated and capable of temperature control (±5°C); the bath should be vented into a hood.
9.6 Silica Gel 60
9.7 Vials - 2 mL glass clear, with Teflon-lined screw cap
9.8 pH indicator paper - close range (0-6.0) and (7.0 - 14.0); wide range (1.0 - 12.0)
9.9 Syringe - 1000µL
9.10 Graduated cylinder - Glass, Class A, 1000 mL
9.11 Pasteur pipette - length 9” and 5-3/4”
9.12 Pipette bulb
9.13 Aluminum foil - heavy duty
9.14 Nitrogen tank - equipped with pressure regulator
9.15 Ultrasonic Disrupter – capable of 300watts output, set on 10 Full power, pulse mode of 50%
9.16 A HP GC system, equipped with a flame ionization detector (FID), is used for analyzing extracts for all target analytes.
9.17 A Restek capillary column (RTX-5, 30m x 0.32mm x 0.25um) is used for analysis.
9.18 HP Chemstation Datasystem is used for data collection, detecting and storage.
9.19 Autosampler vials and caps appropriate to the sample tray are used for sample injection.
9.20 Microsyringes suitable for aliquoting 1.0 µL to 1000 µLs are used for standard preparation and sample dilution.
9.21 Class A volumetrics ranging from 1.0 ml to 250 mls are used for standard, spike and surrogate preparation.

10. Standards and Reagents

10.1. The laboratory’s LIMS system allows for complete documentation and for the traceability of reagents and standards used within the laboratory. The following information relates to the specific reagents and standards used for the performance of the method:

10.1.1. ASTM Type II Water.
10.1.2. Sodium Hydroxide Solution - (10N), Weigh 400 g NaOH into a 1L volumetric and cover with less than 1L reagent water. Use extreme caution when performing this step. Swirl the beaker until all pellets are dissolved (a stir plate can be used to mix the solution). This mixture gets very hot. Let stand until cool. Bring the solution up to the 1L mark with reagent water. Transfer to a 1-liter volumetric flask with several rinses of reagent water and dilute to 1 liter with reagent water. Transfer to a 1000-mL Teflon container.
10.1.3. Sodium Sulfate - Granular, anhydrous, trace pure 10 - 60 mesh placed in a Pyrex pan and heated at 400°C overnight (minimum 4hrs), removed and cooled . Once cooled place in a labelled glass amber jar.
10.1.4. Silica Gel 60 - Granular, anhydrous, trace pure 70-230 mesh. Weigh 60g in a 250 mL glass amber jar and add 1ml DI water to deactivate. Stored at room temperature.
10.1.5. Glass Wool – Pre-rinse all glass wool used during the extraction process with Methylene Chloride.
10.1.6. Sulfuric Acid Solution - (1:1), slowly add 500 mL of Sulfuric Acid to 500 mL of reagent water in a
10.1.7. 1000 mL pyrex container. This mixture will get very warm. Allow to cool before use.

10.1.8. Extraction Solvent - Methylene Chloride (Dichloromethane (Please read SOP-336 before using this solvent in our laboratory)- Omnisolv - suitable for spectrophotometry and gas chromatography (JT Baker) or equivalent.

10.1.9. Carbon Disulfide– (Omnisolv - suitable for spectrophotometry, liquid chromotography and gas chromatography (JT Baker) or equivalent.

10.1.10. Verify the amount of surrogate/spike to add to the sample prior to addition. It can change if a different detection limit is required or the volume of sample being analyzed changes.

10.2. Stock Standards: Are non-Neat standards, received from vendors. These standards are used as intermediate standards to prepare working level standards. For unopened standards, if there is no expiration date assigned by the vendor, the expiration date must be assigned as 1 year from the date of receipt. For open stock standards, the expiration date is 6 months from the date the ampoule is opened or the vendor expiration date, whichever comes first. The following standards are used for the extraction and analysis of Petroleum Hydrocarbons:

Vendor	Catalog #	Description/Conc	Used for Preparation of:
Restek	31097	o-Terphenyl (OTP) 10,000 ug/ml	Curve & Surrogate Soln.
Restek	31096	2-Fluorobiphenyl (2FBP) 10,000 ug/ml	Curve & Surrogate Soln.
NSI	UST-100-08	New Jersey Petroleum Range Mix 17 comps. @ 2.0mg/ml each (total conc..=34,000ug/mL)	Spike Solution.
NSI	C-443-13	Florida TPH Mix 2000 ug/ml ea	Curve Solution
NSI	UST-100-08	New Jersey Petroleum Range Mix 17 comps. @ 2.0mg/ml each (total conc..=34,000ug/mL)	Second Source Standard

NOTE: The FL-PRO Petroleum Range Mix used for the preparation of the Spike Solution should always be a different Lot# from the Curve Standard.

10.3 Working Standards

10.3.1 Are standards made from Neat or from stock standards, and are intended for analytical runs. The expiration date for these standards is 6 months from the date of preparation or the expiration of the parent stock, whichever date is first.

10.3.2 Follow analytical judgement when using standards. Evaluate standards on a daily basis versus past standards and instrument performance. A standard may evaporate or breakdown if proper storage processes are not used. Therefore, standards may have to be discarded before expiration dates.

11. Sample Collections, Preservation, Shipment, and Storage

11.1. Quality Systems SOP QS10 related to Sample Receipt, Handling, & Processing provides details for collection, preservation, shipment, and storage.

11.2. Water samples are preserved with sulfuric acid to pH <2 and cooled to 4°C. Soils are stored at 4°C. Waters must be extracted within 7 days and soils within 14 days from collection and analyzed within 40 days of extraction. Extracts are kept at 4°C. Observe all safety guidelines when handling samples and extracts.

12. Quality Control

12.1 An extraction batch must contain of no more than 20 client samples.

12.2 One BLK1, a BS1, BSD1, and a MS, MSD must be extracted in each batch.

12.3 Please follow guideline from Table 2 for meeting QC criteria.

12.4 All surrogates must pass the established laboratory criteria.

12.4.1 With samples requiring high level dilutions due to matrix interference or due to the abundance of target analytes, the surrogate will be diluted out and no recovery will be recorded. These samples can be reported.

12.4.2For samples failing surrogate recovery high biased due to matrix interference, document the recoveries and notify the supervisor. In most cases, a Case Narrative should be filled out, the client should be notified, and the sample should be reported without a re-extraction. For samples failing the surrogate recovery (OTP) low biased, a re-extraction may need to be performed – check with supervisor. Any low recovery for surrogates reported to client must be noted in case narrative and a CAR must be filled out. This is on a case by case basis and at the discretion of the department supervisor.

13. Calibration and Standardization

13.1Quality Systems SOP QS08 "Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures" related to Calibration Procedures provides laboratory wide protocols for calibration and standardization. See section 14.3 of this SOP.

14. Procedure

14.1 Aqueous Extraction: All waters have a seven-day holding time. Determine the samples necessary to extract from the following sources. Note: never extract samples of unknown origin without discussion with supervisor):

14.1.1 Each day a print backlog from LIMS indicating sample numbers with the respective analysis required

14.1.2 Samples requiring RUSH turn around time may be logged in throughout the day which will require your immediate attention. Log-in personnel and or PM will generally communicate this need.

14.1.3 Periodically check LIMS throughout the day to determine what new samples have arrived. If an analysis is ongoing, extra QC may be avoided by picking up those extractions on the same day.

14.1.4 Batch samples together in the LIMS, and print the bench sheet for the batch. Make sure appropriate number of BLK1, BS1, BSD1, MS1, and MSD1 are listed. From the beginning until the end of the extraction process, continue to fill in pertinent information into the LIMS system.

14.1.5 Wearing lab coat, gloves and safety glasses, get samples from refrigerator. Samples must be signed out of the walk-in refrigerator. Enter the sample numbers, your initials and the date and time removed on the log provided. Inspect as to whether they are in glass amber jars and have a Teflon lid. Find out if any special dilutions are needed for the client. Routine procedures for difficult matrices are listed below.

14.1.6 BAD MATRIX – for example a liquid that is partially sediment, see your supervisor to find out what dilution, if any should be made.

14.1.7 Verify the ID and amount of surrogate/spike to add to the batch prior to addition. It can change if a different detection limit is required or the volume of sample being analyzed changes. Set out the surrogate/spike at least ten minutes before use to allow it to warm to room temperature.

14.1.8 Set up enough separatory funnels to extract the number of samples you have plus any additional spikes and a method blank. A BLK1, BS1, and a BSD1 must be processed with each batch of samples.

14.1.9 Place an Avery label on each separatory funnel containing the Lab #.

14.1.10 Pre-rinse all glassware with Methylene Chloride. Dispose this rinsate into the waste Methylene Chloride reservoir after each rinse. The lab batch code is generated by LIMS. The BLK1 and BS1 label should include all lab #s in this set of samples.

14.1.11 Mark the amber glass container of each sample at the water meniscus with "white out" or with a sharpie for later determination of sample volume. Determine the initial pH of sample and record on extraction sheet. If needed, adjust pH to between 1.0 and 2.0.

14.1.12 ACID pH Adjusting: Adjust the pH to between 1.0 and 2.0, using 1:1 H_2SO_4. Add the acid solution to each sample, spike and method blank. Stopper and shake to insure that pH throughout the sample is changed. Check the pH using a 9"pipette with short-range pH paper. Compare the color to the chart on the pH paper. If the color is not within range add more H2SO4 solution in small increments, as required to attain the proper pH. If sample is received without proper acid preservation, pH adjustment details must be recorded in LIMS.

14.1.13 Using the 1000-mL glass graduated cylinder measure 1000 mL of DI water and transfer it to a separatory funnel for each BLK1, BS1 & BSD1. Transfer sample to separatory funnel that corresponds to the lab # on the sample bottle. Rinse the sample bottle about 3-5 times with 10 mls aliquots of Methylene Chloride. Transfer this rinsate into the separatory funnel labeled with the sample ID.

14.1.14 Fill the sample bottle up to the mark with regular water. Now pour the water into a 1000 mL graduated cylinder. The volume measured is the initial volume to be documented for the sample in the LIMS.

Add appropriate amount of spike to BS1, BSD1, MS & MSD. Also, add surrogate to all samples, BLK1, BS1, BSD1, MS & MSD.

NOTE: If using a syringe to add spike and surrogate, be sure to invert syringe and eliminate air bubble when obtaining surrogate solution and spiking solution. Add solution below the surface of the sample. Someone must verify that the spike and surrogate has been added by placing a check mark on the extraction sheet (& initialing the extraction sheet) as it is added.

14.1.15 Add 50 mL of Methylene Chloride to each sample and to all the batch QC. Shake the sep funnel twice and vent into the hood. Repeat this venting process 3-4 more times and then manually shake the sep funnel for two minutes. Vent the sep funnel at the end of the two minutes. Some samples may require additional venting due to excess pressure buildup. Please use precaution with highly volatile and reactive samples. Place sep funnel, inverted, in shaker apparatus with stopcock open for 3 minutes.

14.1.16 Allow the sample to sit for 10 minutes, if necessary, after it has been shaken. It will separate into two layers with the solvent layer on the bottom. Drain the bottom organic layer into a labeled 250 ml glass beaker first passing the extract through a funnel with glass wool and baked sodium sulfate all pre-rinsed with Methylene Chloride.

14.1.17 Follow Steps 14.1.15 and 14.1.16, two more times with 40 mLs of methylene chloride using the automatic shaker. Collect the extract from this step into the same beaker.

14.1.18 Transfer the extract to a pre-rinsed zymark tube by first passing through a funnel with glass wool and baked sodium sulfate all pre-rinsed with methylene chloride. After pouring the extract into the zymark tube, rinse the collection beaker 3-5 times with Methylene Chloride and transfer the rinsate to the zymark tube. Finally rinse the funnel with an adequate amount of Methylene Chloride using a Teflon squirt bottle. This ensures optimum transfer of all compounds of interest. Now concentrate the extract to 1.0 mL using the turbovap concentrator.

14.1.19 Turbo-Vap Operation: Adjust the pressure of nitrogen gas tank to 30 psi. Make sure the tank has 200 psi or more on the main valve. The temperature of the bath should be approximately 45-50°C. The pressure target range should be about 15-20 psi. Place the glass evaporator tube in the Turbo-Vap. Be sure to push tube down so the tip slides into the sensor well. Close the lid to start concentration. Check that each position with a tube has an orange light showing. If the orange light is not steady, bubbles may be detected by the sensor and need removal. (See Turbo-Vap manual).

14.1.20 When the beep sounds indicating the end of concentration, the extract will be at approximately 1 mL Remove the tube from the bath.

14.1.21 Add methylene chloride to dissolve any precipitate. Transfer extract to a 4.0 ml vial, rinsing with methylene chloride. Adjust volume with methylene chloride to 2 ml. Add 0.3 g of silica gel and shake for 5 min.

14.1.22 Sign the batch into the extraction laboratory Hobart. Refrigerate at 4°C or carry directly to the instrument operator. Remit custody of the batch to the analyst or technician. The extract is now ready to be analyzed.

14.1.23 The extraction is now complete. Clean all glassware used during the extraction and store appropriately. Please refer to the glassware cleaning SOP for additional guidance.

14.2 Solid Extraction (may also follow extraction procedure outlined in SOP 343). All solids have a fourteen-day holding time counted. Determine the samples necessary to extract from the following sources (Note: never extract samples of unknown origin without discussion with supervisor):

14.2.1 Each day a print backlog from LIMS indicating sample numbers with the respective analysis required

14.2.2 Samples requiring RUSH turn around time may be logged in throughout the day which will require your immediate attention. Log-in personnel and or PM will generally communicate this need.

14.2.3 Periodically check LIMS throughout the day to determine is new samples have arrived. If an analysis is ongoing, extra QC may be avoided by picking up those extractions on the same day.

14.2.5 Batch samples together in the LIMS, and print the bench sheet for the batch. Make sure appropriate number of BLK1, BS1, BSD1, MS1, and MSD1 are listed. From the beginning until the end of the extraction process, continue to fill in pertinent information into the LIMS system.

14.2.6 Wearing lab coat, gloves and safety glasses, get samples from refrigerator. Samples must be signed out of the walk-in refrigerator. Enter the sample numbers, your initials and the date and time removed on the log provided. Inspect as to whether they are in glass jar. Routine procedures for difficult matrices are listed below:

BAD MATRIX – for example a solid that is partially oil, see your supervisor to find out what dilution, if any should be made. Verify the ID and amount of surrogate/spike to add to the batch prior to addition. It can change if a different detection limit is required or the volume of sample being analyzed changes. Set out the surrogate/spike at least ten minutes before use to allow it to warm to room temperature.

14.2.7 Get out enough 250mL beakers to extract the number of samples you have plus any additional spikes and a method blank. A BLK1, BS1, and a BSD1 must be processed with each batch of samples. A matrix spike and a duplicate or a matrix spike duplicate must be processed for each extraction batch (up to a maximum of 20 samples). If sufficient sample is not available to perform a batch MS & MSD indicate such on the extraction sheet.

14.2.8 Pre-rinse all glassware with Methylene Chloride. Dispose this rinsate into the waste Methylene Chloride reservoir after each rinse. Label each 250mL beaker with the Lab ID.

14.2.9 Pre-weigh beakers and tare. Weigh 25g aliquot of the sample to the beaker and record weight to nearest 0.01g in extraction log. Add 25g dried Sodium Sulfate powder and stir the mixture well with a stainless steel spatula to a free-flowing sandy texture. If sample mixture forms large clumps, add more Sodium Sulfate to achieve proper texture (note in extraction log).

14.2.10 *It is extremely important that waste (when appropriate), soil and sediment samples be mixed thoroughly to ensure that the sample is as representative as possible of the sample media. The most common method of mixing is referred to as quartering. The quartering procedure should be performed as follows:*

14.2.10. The material in the sample pan(inorganic-plastic/organic-aluminum) should be divided into quarters and each quarter should be mixed individually.

14.2.10.2 Two quarters should then be mixed to form halves.

14.2.10.3 The two halves should be mixed to form a homogenous matrix. This procedure should be repeated several times until the sample is adequately mixed.

NOTE: Samples that are clay type materials should be handled in a different manner. Due to these type sample matrices having an affinity to stick to most anything that touches it, another approach must be followed. Obtain a representative sub-sample aliquot from the center or middle section of the sample container.

14.2.11 Add appropriate amount of spike to BS1, BSD1, MS & MSD. Also, add surrogate to all samples, BLK1, BS1, BSD1, MS & MSD.

NOTE: If using a syringe to add spike and surrogate, be sure to invert syringe and eliminate air bubble when obtaining surrogate solution and spiking solution. Add solution below the surface of the sample. Someone must verify that the spike and surrogate has been added by placing a check mark on the extraction sheet (& initialing the extraction sheet) as it is added.

14.2.12 Add 60 mL of Methylene Chloride to each sample and to all the batch QC. Sonicate each sample for 3 minutes in Ultrasonic Disruptor (set on 10 Full power – pulse mode) at a pulse rate of 50%.

14.2.13 Decant the Methylene Chloride extract through a funnel with glass wool and baked sodium sulfate all pre-rinsed with Methylene Chloride, into a rinsed zymark tube.

14.2.14 Follow Steps 14.2.12 and 14.2.13, one more time with 60 mLs of methylene chloride. Collect the extract from this step into the appropriately labeled tube.

14.2.15 After pouring the extract into the zymark tube, rinse the beaker 3-5 times with Methylene Chloride and transfer the rinsate to the zymark tube. Finally rinse the funnel with an adequate amount of Methylene Chloride using a Teflon squirt bottle. This ensures optimum transfer of all compounds of interest. Now concentrate the extract to 1.0 mL using the turbovap concentrator.

14.2.16 Turbo-Vap Operation: Adjust the pressure of nitrogen gas tank to 30 psi. Make sure the tank has 200 psi or more on the main valve. The temperature of the bath should be approximately 45-50°C. The pressure target range should be about 15-20 psi. Note the turbovap pressure and temperature on the extraction logbook.

14.2.17 Place the glass evaporator tube in the Turbo-Vap. Be sure to push tube down so the tip slides into the sensor well. Close the lid to start concentration. Check that each position with a tube has an orange light showing. If the orange light is not steady, bubbles may be detected by the sensor and need removal. (See Turbo-Vap manual).

14.2.18 When the beep sounds indicating the end of concentration, the extract will be at approximately 1 mL. Remove the tube from the bath.

14.2.19 Add methylene chloride to dissolve any precipitate. Transfer extract to a 4.0 ml vial, rinsing with methylene chloride. Adjust volume with methylene chloride to 2 ml. Add 0.3 g of silica gel and shake for 5 min.

14.2.20 Sign the batch into the GC laboratory Hobart. Refrigerate at 4°C or carry directly to the instrument operator. Remit custody of the batch to the analyst or technician. The extract is now ready to be analyzed.

14.2.21 The extraction is now complete. Clean all glassware used during the extraction and store appropriately. Please refer to the glassware cleaning SOP for additional guidance.

14.3 GCFID Analysis

14.3.1 Quality Systems SOP QS09 “General and Commonly used Laboratory Calculations” provides details on general calculations used throughout the laboratory.

14.3.2 Follow guidelines provided in the method for GC-FID conditions and sample volume to be injected for method FL Pro.

14.3.3 It is recommended that a solvent Blank be analyzed at the beginning of every sequence to ensure that the analytical instrument is free of contaminants.

14.3.4 All extracts within a batch are run on the Instrument after meeting calibration criteria as described in Table 2.

14.3.5 Qualitative and quantitative analysis is performed on samples.

14.3.5.1 Qualitative Analysis for specific carbon ranges or fuel patterns, such as; mineral spirits, kerosene, JP-4 and heavy oils are performed per client request compared to specific standards. (See Table 3)

14.3.5.2 Quantitative Analysis is performed using the following tools:

14.3.5.2.1Retention times for the range FL PRO C8-C40 are set daily using the mid-level of the calibration (if applicable) or the first CCV of the run by subtracting 0.05min. from the RT of C8 and adding 0.05min. to the RT of C40. FL PRO analysis is performed by running 6 calibration levels of a TPH mix from C-8 through C-40 (17 peaks). A response factor is calculated for each calibration standard (amount sum of 17 peaks/ std amount * 17), then an Average Response factor is calculated for all 6 standards. This Average Response Factor is put in the method for uncalibrated peaks. Percent RSD must be less than or equal to 20%.

14.3.5.2.2Surrogates o-Terphenyl and 2-fluorobiphenyl are added to each calibration standard at the same concentration. Initial calibration must pass acceptance criteria in Table 2.

14.3.5.2.3 Analyte concentration must be within the calibration curve range. If the analyte concentration exceeds the calibration curve range, the extract must be diluted & rerun to bring the concentration within the calibration range. Use the calculation in section 15 to report the final results for the sample.

14.3.5.2.4 Target analytes are calculated using the calibration curve and by incorporating any adjustments for inital or final volume and dilutions.

15. Data Analysis and Calculations

15.1 Quality Systems SOP QS09 "General and Commonly used Laboratory Calculations" provides details on general calculations throughout the laboratory.

15.2 Calculate the calibration factor for each analyte at each concentration as:

$$CF = \frac{\text{Peak Area (or Height) of the Compound in the Standard}}{\text{Mass of the Compound Injected (in nanograms)}}$$

The mean CF is calculated as follows:

15.3 The standard deviation (SD) and the relative standard deviation (RSD) of the calibration factors for each analyte are calculated as follows:

$$SD = \sqrt{\frac{\sum_{i-1}^{n}(CFi - \overline{CF})^2}{n-1}}$$

$$RSD = \frac{SD}{AvgCF} X\ 100$$

15.4 Calibration verification involves the calculation of the percent drift (linear or quadratic) or the percent difference (average) of the instrument response between the initial calibration and each subsequent analysis of the verification standard. Use the equations below to calculate % Drift or % Difference, depending on the calibration procedure used.

$$\%\ Drift = \frac{(\text{Calculated concentration} - \text{Theoretical concentration})\ x\ 100}{\text{Theoretical Concentration}}$$

where the calculated concentration is determined from the initial calibration and the theoretical concentration is the concentration at which the standard was prepared.

$$\%\ Difference = \frac{(CCV\ CF - Average\ CF)\ x\ 100}{Average\ CF}$$

15.5 External standard calibration - The concentration of each analyte in the sample may be determined by calculating the amount of standard injected, from the peak response, using the calibration curve. The concentration of a specific analyte is calculated as follows:

Aqueous Samples**:**

$$\text{Concentration } (\mu g/L) = [(A_s)(V_t)(D)]/[(CF)\overline{(V_i)}(D)]$$

where:

A_s = Response for the analyte in the sample, units may be in area counts or peak height.

V_t = Total volume of sample, mL.

D = Dilution factor, if dilution was made on the sample prior to analysis. If no dilution was made, D = 1.

$\overline{CF}$ = Mean calibration factor from initial calibration (area/ng)

V_i = Volume of extract injected, µL.

V_s = Volume of aqueous sample , mL.

Using the units specified here for these terms will result in concentration units of ng/mL, which is µg/L.

Nonaqueous Samples**:**

$$\text{Concentration } (\mu g/kg) = [(A_s)(V_t)(D)]/[(CF)(V_i)\overline{(W_s)}]$$

where:

W_s = Weight of dry sample extracted, g.

A_s, V_t, D, $\overline{CF}$ and V_i have the same definition as for aqueous samples.

16. Method Performance

16.1 Demonstration of Capability (DOC): Each analyst must perform a DOC prior to reporting data. The analyst must prepare (for prep technicians) and analyze (analysts reviewing and reporting data) 4-LCS samples. The data is calculated for accuracy and precision requirements. The DOC form, as listed within section 2.5 of the Quality is completed by each analyst and then provided to the supervisor for further processing and approval.

16.2 See method FL-PRO for method performance.

17. Pollution Prevention

17.1 Quantity of chemicals purchased should be based on expected usage during its shelf life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

18. Data Assessment and Acceptance Criteria for Quality Control Measures

18.1 Quality Control SOP QS05, “Data Deviations/Interpretations/Exceptions: Laboratory Non-Conformance/ Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results,” provides details on data assessment and acceptance criteria for Quality Control Measures. Table 2 of this SOP provides information on QC samples, frequency, and the associated criteria specific to the performance of this method.

19. Contingencies for Handling out-of-control or unacceptable data

19.1 Quality Control SOP QS05, “Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results”, provides details on handling out of control data. Table 2 within this SOP also lists corrective actions associated with the failure of the various QC samples employed for the performance of this method.

20. Waste Management and Pollution Prevention

20.1 Please see Waste Disposal SOPs 210 and 405 for proper waste disposal.

20.2 Quantity of chemicals purchased should be based on expected usage during it’s shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

21. References

21.1 Method for Determination of Petroleum Range Organics (Method FL-PRO)

22. Tables, Diagrams, Flowcharts and Validation Data

TABLE 1 – Analytes, Reporting Limit (RL), & Low Calibration Standard				
Parameter	DL	LOD	LOQ/RL	Low Cal
FL-PRO	0.085ug/L	0.16ug/L	0.34ug/L	0.17ug/L
FL-PRO	5.6ug/Kg	10.7ug/Kg	22.6ug/Kg	11.3ug/Kg

Table 2 - Method Quality Control Requirements Summary			
QC Check	**Minimum Frequency / Requirements**	**Acceptance Criteria**	**Corrective Action for Failures / Data Useability**
Calibration Curve	• Prior to analyzing any samples • A minimum of 5-points for linear fits • A minimum of 6-points for quadratic fits • Low standard at the RL/LOQ level	• Linear correlation coefficient of at least 0.995 • Quadratic squared correlation coefficient of at least 0.99 • Average CF =< 20% RSD • Manual integrations on curve standards must have supervisory approval • Must follow curve processing requirements from SOP QS08	• Re-evaluate curve mix and makeup • Re-run curve • Check instrument for maintenance needs • Re-prep the curve standards Samples cannot be analyzed until there is a passing calibration
ICV	Alternate source standard to be analyzed after every calibration curve	• $\leq$ 25% drift or difference for all analytes	• Re-analyze an ICV from a different source • Re-prep and re-analyze the ICV • Re-calibrate and verify standard preps and sources
CCV	• At the beginning of every sequence • For every 10-client samples and at the end of the sequence • The concentration must be varied from low to mid range	• $\leq$ 25% drift or difference for all analytes	• Evaluate the system for required maintenance • Obtain passing CCV • Reanalyze all samples injected since last passing CCV • Q-qualify if reanalysis is not possible
MB	One per prep batch	• Must be less than ½ the RL/LOQ or <1/10th any sample concentration or <1/10th the regulatory limit – whichever is greater.	• Re-analyze to confirm the positive value • If MB results are between the LOD and RL/LOQ, assess the data and notify the PM for possible further action • Re-extract affected samples associated with the MB • NCR and final report qualification will be required for affected samples if re-extraction is not possible.

Table 2 - Method Quality Control Requirements Summary			
QC Check	**Minimum Frequency / Requirements**	**Acceptance Criteria**	**Corrective Action for Failures / Data Useability**
Surrogates	Spike in every field or QC sample and standard	Surrogate / Water / Soil OTP: 82-142 (method), 30-140 (in-house) / 62-109 (method), 45-135 (in- house) 2-FBP: 50-150 (default) / 50-150 (default) Note: Project limits will be used when specified.	• Batch QC should pass method limits. • Reanalyze to confirm recovery if failing in-house limits. • Re-extract associated samples, if still failing in-house limits. • Q-qualify if re-extraction is not possible or verifies exceedence.
LCS	One per prep batch	Water 55-118% Soil 63-143% Note: Project limits will be used when specified.	• Reanalyze to confirm recovery. • Re-extract associated samples, if still failing • Q-qualify if re-extraction is not possible
LCSD	One per prep batch, when MS/MSD not included.	Water 55-118% RPD ≤20% Soil 63-143% RPD ≤25% Note: Project limits will be used when specified.	• See LCS
MS/MSD	One per prep batch, if sample volume available.	Water 41-110% RPD ≤20% Soil 51-215% RPD ≤25% Note: Project limits will be used when specified.	• Reanalyze to confirm recovery. • Re-extract if failure is judged to be due to extraction/analysis. • J-qualify associated parent sample if reporting from results exceeding limits.
DOC Study	• Initially per analyst prior to reporting data • Annually • Follow specific guidelines from section 16 for the preparation and analysis of DOC samples	• Must meet the criteria of the LCS for average recovery • Precision criteria is 20% standard deviation.	• Re-prep and / or • Re-analysis
LOD Verification	Every quarter	• Parameter must be detected with response 3x the noise level	• Re-prep and / or re-analysis • Raise concentration
LOQ Verification	Every quarter	• Bias Requirement: Organics 50-150% • The LOQ value must be greater than the LOD value	• Re-prep and / or re-analysis • Raise concentration

Table 2 - Method Quality Control Requirements Summary

QC Check	Minimum Frequency / Requirements	Acceptance Criteria	Corrective Action for Failures / Data Useability
Retention Time Study	• Prior to running samples • With major instrument changes	3 injections over 72 hours – calculate standard deviation of the measured retention times. Windows +/- 3xSD	If <0.05minutes use +/0.05 minutes.

Table-3

Qualitative Analysis Tool

SOP Title: **BNA & Pesticide/PCBs & TPH NON-AQUEOUS MATRIX (MICROWAVE EXTRACTION) USING SW-846 METHOD 3546**

SOP NUMBER: **SOP-343**

REVISION NUMBER: **0**

APPROVED BY:

SECTION MANAGER

QUALITY ASSURANCE OFFICER

EFFECTIVE DATE: **08/01/09**

DATE OF LAST REVIEW: **08/01/09**

BNA & Pesticide/PCB & TPH NON-AQUEOUS MATRIX
(Microwave Extraction)
Using SW846 METHOD 3546

1. SCOPE AND APPLICATION

a. This SOP describes the extraction of BNAs, pesticides/PCBs, and TPHs from soil, sediment, sludges and waste solids by an automated method (3546).

2. SUMMARY

a. Soil and solid samples are mixed with sodium sulfate and extracted with solvent in a Microwave extractor for BNAs, Pesticides/PCBs, or TPHs. The extracts are then concentrated by a Turbo Vap concentrator.

3. INTERFERENCES

a. Solvents, reagents, glassware, and other sample processing apparatus can add interferences to sample analysis. Method blanks must be extracted under the same conditions as samples to demonstrate freedom from interferences.
b. Phthalate esters commonly found in plastics can interfere with the analysis. Plastics should be avoided.
c. Soap residue can degrade certain analytes such as aldrin and heptachlor. Glassware should be solvent rinsed to avoid this problem.

APPARATUS AND MATERIALS

d. Stainless Steel spatula
e. Microwave extractor unit with 40 position carousel, electronic components, and ample ventilation
f. Microwave extraction Teflon tubes, capacity approximately 75mL
g. Suitable Teflon cap and screw-top lid
h. Drying column (Chromatographic column) – 20mm I.D. x 300mm
i. Vial – 2mL clear with Teflon-lined screw cap
j. Vial – 12mL clear with Teflon-lined screw cap
k. Syringe – 1mL, 500uL
l. Pasteur pipet – 9” length
m. Pasteur pipet bulb
n. Labels – Dymo
o. Aluminum foil – heavy duty
p. Nitrogen tank – equipped with pressure regulator
q. TurboVap Concentrator with 200mL concentrator tubes
r. Teflon funnels for pouring off
s. Balance – capable of weighing to 0.1grams
t. Aluminum pie pans for mixing samples
u. Filter paper – 185mm

4. REAGENTS

a. Sodium Sulfate ($Na2SO4$) – Granular, anhydrous, trace pure 10-60 mesh (purchased in bulk containers from Fisher #S415-10S or equivalent)
b. Methylene Chloride (Please read SOP – 336 before handling this solvent in our laboratory) (Dichloromethane) – suitable for spectrophotometry and gas chromatography (Fisher #D151-4 or equivalent)
c. Hexane – suitable for spectrophotmetry and gas chromatography (Fisher #H303-4)
d. Surrogate/Spike Solutions – Verify the amount of surrogate/spike to add to the sample prior to addition. It can change if a different detection limit is required or the volume of sample being analyzed changes or if the initial concentration of stock is different than that listed below:
    i. **BNA Surrogate (100ug/mL)** – The base neutral and acid surrogates are mixed together in one solution. This solution is prepared in methanol by making a dilution of stock purchased from a reputable vendor. Use 0.5mL of this solution per 15g of non-aqueous sample. **(For low-level PAHs use 1.0mL of 1.0ug/mL BN Surrogate spiking solution.)**
    ii. **BNA Spiking Solution #1 & #2 (100 ug/mL)** – The base neutral and acid spiking solutions are mixed together in one solution. This solution is prepared in methanol by making a dilution of stock purchased from a reputable vendor with same compounds as for calibration. Use 0.5 mL of this solution per 15g of non-aqueous sample. **(For low-level PAHs use 1.0mL of 1.0 ug/mL PAH spiking solution.) The BNA Spiking solutions contain all targets that are calibrated for GC/MS. DOD QSM requires all targets to be spiked in the LCS and MS/MSD.**
    iii. **TCMX/DCB (2,4,5,6-Tetrachloro-meta-xylene/Decachlorobiphenyl) Surrogate solution is** prepared in acetone by making a cut on stock purchased from a reputable vendor. 0.5mL at 0.5 ug/mL of this solution is added per 15g of non-aqueous sample.
    iv. **PCB Spiking Solution** – Arochlor 1016/1260 or the PCB of choice (1242, 1248, 1254, or 1260 are the most common) is prepared in acetone at a concentration of 5.0ug/mL. PCB stock is usually purchased from RESTEK or equivalent. The PCB to use may be determined by viewing historical data or asking the GC operator. Use 0.5mL per 15.0g of non-aqueous sample.
    v. **Pesticide Spiking Solution** – A spiking solution is prepared at 1.0 ug/mL. Use 0.5mL per 15g of non-aqueous sample.
    vi. **TPH Surrogate** – Surrogate solution is prepared in acetone by diluting stock ortho-terphenyl standard to a final concentration of 20 ug/mL. Use 1mL per 15 grams of sample.
    vii. **TPH Spike** – A spiking solution is prepared by extractions analyst that has a concentration of 1000 ug/mL in acetone.

5. SAMPLE COLLECTION, PRESERVATION, AND HOLDING TIMES

a. Samples are collected in an appropriate size wide-mouth glass jar (4oz. or 8 oz.) with a Teflon-lined cap.
b. Samples are preserved by cooling to 4°C.
c. Holding time is 14 days from collection date to extraction.

6. PROCEDURE

a. All soils have a 14-day holding time counted from the day they are sampled. Determine the samples necessary to extract using the following information (DO NOT extract samples for which you have no information.):
    i. Each day a backlog is generated in ELEMENT providing all relevant sample information, including samples numbers and respective analysis required.
    ii. Samples requiring RUSH turn around time may be logged in throughout the day which will require your immediate attention. Log-in personnel will generally communicate this need.
    iii. Check the backlog throughout the day to re-evaluate priority if needed.
b. Wearing lab coat, gloves, and safety glasses, get samples from cooler. Samples must be signed out of the walk-in refrigerator. Enter the sample numbers, your initials, and the date and time removed on the log provided. Inspect as to whether they are in glass and have a Teflon lid. Find out if any special dilutions need to be made for this client. If the sample has a particularly bad matrix or a strange matrix, see your supervisor to find out if a microwave extraction is truly necessary.
c. Get twice the number of aluminum pie pans to prepare the number of samples you have plus any additional spikes of LCSs and a method blank. A method blank and LCS must be processed with each set of samples. A matrix spike, a duplicate or a matrix spike duplicate and a LCS must be processed for each analytical batch (up to a maximum of 20 samples). Using the LIMS, create a batch of samples and print off sample labels. The LIMS will create a unique batch sequence number.
d. Decant and discard any water layer on a sediment sample by carefully pouring this off into a trashcan.
e. Dump the entire sample into an aluminum pie pan and mix sample thoroughly with a spatula until mixture is homogenous. Discard any foreign objects such as sticks, leaves, and rocks.

It is extremely important that waste (when appropriate), soil and sediment samples be mixed thoroughly to ensure that the sample is as representative as possible of the sample media. The most common method of mixing is referred to as quartering. The quartering process should be performed as follows:

- *The material in the sample pan (inorganic-plastic/organic-aluminum) should be divided into quarters and each quarter should be mixed individually.*
- *Two quarters should then be mixed to form halves.*
- *The two halves should be mixed to form a homogenous matrix.*

This procedure should be repeated several times until the sample is adequately mixed.

NOTE: Samples that are clay type materials should be handled in a different manner. Due to these type sample matrices having an affinity to stick to most anything that touches it, another approach must be followed. Obtain a representative sub-sample aliquot from the center or middle section of the sample container

f. Place an aluminum pie pan on the balance and zero it. Calibrate balance with ASTM class-1 Troemner weights or equivalent, bracketing desired weight (50g, 20g, 10g, 5g, 1g). Record calibration in the Extraction calibration/temperature logbook. Using a spatula, transfer the **appropriate weight, {10-20 grams depending upon client or project specific Detection Limits (DL) and/or Reporting Limits (RL)}**, of a representative sample to the nearest 0.1 gram. Normally 10 or 15g sample weights are used. Record this amount on your label. Put your label on the side of the 400-mL beaker. For spiking purposes, weigh 3 aliquots of the appropriate sample. Pick a sample with a good matrix, one that mixes well, non-oily, etc.
g. Add ~ 15 grams of sodium sulfate to the aluminum pie pan. Using a spatula and/or a glass rod, mix the sample thoroughly with the sodium sulfate until it becomes a sandy texture. If necessary, add additional sodium sulfate. When removing the spatula or glass rod from the mixed sample, leave behind all the sample possible. Cover the aluminum pie pan with foil and continue to weigh up the remaining samples. For the method blank and LCS, weigh up 15 grams of sodium sulfate. The matrix used for the method blank and LCS must be free of the analytes of interest and processed through the same analytical steps as the samples.
h. Verify the amount of surrogate/spike to add to the sample prior to addition. It can change if a different detection limit is required or the volume of sample being analyzed changes. Set out the surrogate/spike at least ten minutes before use to allow it to warm to room temperature. Someone must verify that the surrogate/spike has been added by watching and signing off on bench sheet.

NOTE: Surrogate and spike should be added just prior to extraction.

i. Using the 1-mL glass syringe designated for BNA surrogate, add 0.5 mL of BNA surrogate to each sample, spike, and blank. **(For low level PAHs use 1.0 ml of the 1.0 µg/mL BN Surrogate spiking solution.)** or using the 1.0-mL glass syringe marked TCMX/DCB surrogate, add 0.5 mL of TCMX/DCB surrogate to each sample, blank and spike. TPH samples will need 1.0 mL of appropriate.

   For the BNA sample in each analytical batch selected for spiking, use the 0.5-mL glass syringe marked Base Neutral Acid Spiking to add 0.5 mL of the Base Neutral Acid Spiking solution. **(For low level PAHs use 1.0 ml of the 1.0µg/mL PAH spiking solution.)**
   For Pest/PCB samples, determine if the sample will require a Pesticide Spike and/or a PCB Spike. Proceed as follows:
   Pesticide and PCB - set up two LCS's – one for Pesticide getting an AB MIX spike and one for PCB, which should be spiked with PCB 1660. In addition to the LCSs, a matrix spike/matrix spike duplicate is necessary for the pesticide. Prepare a PCB matrix spike/ matrix spike duplicate if requested by the client.
   Pesticide only – To the sample in each analytical batch selected for spiking, add 0.5 mL of Pesticide Spike (Mix A&B) with a glass syringe dedicated for Pesticide Spike.
   PCB only - To the sample in each analytical batch selected for spiking, add 0.5 mL of PCB 1016/1260 (unless otherwise specified, 1248 for BB&L) using a 1.0 mL glass syringe dedicated to that PCB. Add 20 grams of Na_2SO_4.

j. Place a Teflon cap and Teflon screw top on the Teflon microwave tube. Using the cap tightener station, tighten the caps and invert sample to insure proper mixing and check for leaks in cap.
k. Place microwave tubes in microwave carousel making sure they are in order and spaced evenly throughout the carousel to insure proper heating while in microwave.
l. Place microwave carousel in microwave making sure the carousel is properly lined up with the turning mechanism.
m. Choose saved program option based on total number of samples to extract and begin process by pressing the start button. The program is set to EPA method 3546 specifications.

   For 1-15 samples:
   Max power: 800W 100%
   Ramp time: 15:00
   Control temperature (in Celsius): 110
   Hold time: 10:00
   Cool down: 5:00

For 16-40 samples:
Max power: 1600W 100%
Ramp time: 15:00
Control temperature (in Celsius): 110
Hold time: 10:00
Cool down: 5:00

n. Allow samples to cool in the carousel for an additional 30 minutes before attempting to handle the extracts.
o. Transfer the extract to a pre-rinsed turbo vap tube by first passing through a funnel with P4filter paper sodium sulfate. All tubes and funnels should be pre-rinsed with Methylene Chloride. After pouring the extract into the turbo, rinse the microwave tube 3 times with Methylene Chloride and transfer the rinsate to the turbo. Finally, rinse the funnel with an adequate amount of Methylene Chloride using a Teflon squirt bottle. This ensures optimum transfer of all compounds of interest.
p. Now concentrate the extract to 1.0mL using the turbovap concentrator.
    i. **Turbo-Vap Operation:** Adjust the pressure of nitrogen gas tank to 50 psi. Make sure the tank has 200 psi or more on the main valve. The temperature of the bath should be approximately 50-55°C. The pressure target range should be about 20-25 psi.
    ii. Place the turbo vap tube in the Turbo-Vap. Be sure to push tube down so the tip slides into the sensor well. Close the lid to start concentration. Check that each position with a tube has an orange light showing. If the orange light is not steady, bubbles may be detected by the sensor and need removal. (See Turbo-Vap manual).
    iii. When the beep sounds indicating the end of concentration, the extract will be at approximately 1 mL. Remove the tube from the bath.
q. BNA and TPH samples need to be concentrated to ~1.0mL while Pesticides and PCB should be concentrated to ~5.0mL in turbo vap. Using clean solvent, rinse turbo with Pasteur pipet and bring sample to volume in sample vial.

7. DOCUMENTATION OF CAPABILITY (DOC)

a. Each analyst must perform a DOC to demonstrate proficiency with this method. Refer to SOP-413 for guidance.

8. WASTE MANAGEMENT AND POLLUTION PREVENTION

a. Please see Waste Disposal SOP-405 for the proper disposal of waste generated from this area.
b. Quantity of chemicals purchased should be based on expected usage during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

9. METHOD PERFORMANCE

a. Refer to SOP-201 SOP-211 and SOP-219 for method performance.

10. REFERENCES

a. EPA Methods SW-846, Method 3546

11. DEFINITIONS

a. Refer to SOP-431 for definitions.

12. HEALTH AND SAFETY

a. Wear appropriate personal protection equipment when working with chemicals or samples.
b. Use the lab hoods when working with solvents.
c. Use caution when mixing strong acids or bases. Solutions will become extremely hot when mixing with water. Avoid splashing these solutions so they won't come in contact with the skin or eyes. If this happens, flush with lots of water. Contact your supervisor if serious and medical attention is needed.

LABORATORY SAMPLE RECEIVING,
LOG IN AND STORAGE
STANDARD OPERATING PROCEDURES

SOP NUMBER: **SOP-404**

REVISION NUMBER: **13**

APPROVED BY:

SECTION MANAGER

Randy H. Ward

TECHNICAL DIRECTOR

EFFECTIVE DATE: **06/29/09**

DATE OF LAST REVIEW : **06/29/09**

LABORATORY SAMPLE RECEIVING, LOG IN AND STORAGE

This SOP lists in as much detail as possible our daily procedures for sample receiving, log in and storage of laboratory samples. Keep in mind that there may be project specific requirements that are more strict or different than our routine procedures. In these instances, the project specific requirements must be met and followed. Although a few project specific requirements are detailed in this SOP, i.e. USACE certification issues, not every situation can be addressed. If there is ever any uncertainty on what procedures must be followed, please see the Testing Coordinator immediately. If ever in doubt, always go with the more stringent requirements. This document will constantly be reviewed and revised as necessary.

SAMPLE ACCEPTANCE CRITERIA

A sample may be rejected for compliance purposes if it does not meet the following criteria. Analyses may only proceed after notification and approval to proceed from the client or from the laboratory manager.

1. Sample must be properly preserved and in the proper container for the requested analysis.
2. Sample integrity must be maintained. The container shall be intact without cracks, leaks, or broken seals.
3. Adequate sample volume must be received for the requested analysis, including volume for any requested QA/QC (MS/MSD).
4. The sample ID on the bottle label must match the sample ID listed on the chain of custody.
5. The sample container label and the chain of custody must be completed with indelible ink. The sample label must be intact and list all necessary information; to include: sample date, sample time, sampler, and sample ID/location. The chain of custody shall also indicate sample date and time, requested analyses, and all necessary client information.
6. Sample temperature must be less than 6°C or received on ice.
7. Sample must be within holding time for the requested analysis.

These issues are discussed in more detail below under the "Sample Receiving" section of this document.

I. Sample Receiving

A. Samples are received at the Empirical Laboratories on 621 Mainstream Drive, Suite 270 Nashville, TN 37228.

1. The majority of samples are shipped in coolers by couriers such as Federal Express and UPS. All couriers are generally received in the Empirical Laboratories Sample Receiving (SR) area loading dock in back of the laboratory. The laboratory is located close to the Federal Express (FedEx) distribution station, therefore we do pick up our

coolers at the FedEx location and transport them back directly to the laboratory. Some coolers and/or samples are delivered directly to the SR area by the sampler and/or client.

2. Some coolers and/or samples may be received directly by Empirical Laboratories Sample Receiving personnel. If samples are hand delivered by the client make sure that necessary paperwork is included and that you sign and date the chain of custody, as well as record the temperature of the samples on the chain of custody as well. If the *Empirical Laboratories* ***Chain of Custody [Attachment II***] is used the white and yellow copy of the chain of custody is retained and the pink copy must be given to the client.

B. When going through the required steps for Sample Receiving and Sample Log In, keep in mind that a ***Corrective Action Report (CAR) for Sample Receiving [Attachment III]*** must be completed to document any problems, discrepancies, project changes, etc. encountered during the process. This includes but is not limited to incorrect sample containers, improper preservatives [chemical and temperature], chain of custody discrepancies, sample descriptions, etc. A CAR may be completed just to keep a record of a situation, which is not actually "out of compliance."

1. Make sure that all information on the CAR is stated clearly and very detailed. Many times it is necessary to refer to these documents a year or more after they were completed. Document all correspondence including name, date, company and response.

2. The CAR forms must be numbered starting with No. 001 at the beginning of the year (e.g. 01-001). No two forms should have the same number. All CARs must be forwarded to the Project Manager and/or receiving manager for approval and distribution. THIS MUST BE DONE ASAP OF WHEN THE PROBLEM/SITUATION IS DISCOVERED.

C. Visually inspect all coolers for tampering, custody seals present and intact (if applicable) leakage, etc. If a cooler has been damaged beyond repair, unpack the samples and discard the cooler as to not reuse it. If you suspect a cooler may be damaged or is extremely dirty this cooler must not be reused. If coolers were sent by Federal Express, examine the Federal Express airbills for the number of packages in the shipment and make sure that all the packages (coolers, boxes etc.) in a group have been received. If there are any problems the Project Manager must be contacted immediately. If anything looks unusual, take the time to check it out and document the situation and findings.

D. Open each cooler in order to quickly inspect the contents and to locate the chain of custody. Sample Receiving personnel should wear the following personal protection equipment: gloves, safety glasses and a laboratory coat. All coolers received from projects with the **US Army Corps of Engineering (USACE) and AFCEE** projects should be opened under the hood in the sample storage room. Sign then list the date and time received on the chain of custody. The time received must reflect the actual time the samples were received even though they may be logged into the system at a later time. Samples received on Saturday may be processed on the following Monday morning, or samples received late in the day during the week may be processed the next morning. All cooler(s) must be opened, examined for leakage, breakage etc., the temperature measured and the chain of custody signed and dated to reflect the actual date and time which they were received. The samples must be delivered to the appropriate analytical department or put in cold storage as soon as possible.

1. Attach any shipping receipts, work orders, etc. to the chain of custody.

2. If a chain of custody or other paperwork is not sent, the client must be contacted and the samples temporarily placed on hold in cold storage. In some instances the log-in person may complete a chain of custody. The required information may be found on the sample containers or it may be necessary to call the client to get the missing information (i.e. sample ID, collection date and time, etc.). Note on the chain of custody that it was completed by laboratory personnel and record the name of the person with whom you spoke. All attempts to encourage our customers to complete a chain of custody or submit written information for samples must be made.

3. Project specific paperwork may be required. For all projects, a ***Cooler Receipt Form [Attachment IV]*** must be completed for each cooler received. Sample receiving personnel must begin completing this form as soon as a cooler is received and complete this form as samples go through the log in process.

E. The temperature of each cooler or set of samples must be measured as quickly as possible using a thermometer with 0.1°C increments. This thermometer must be calibrated against a NIST certified thermometer once a year and this information recorded in a bound notebook. The Certificate of Calibration for the NIST thermometer is kept on file at the QAO's desk. The thermometer must be tagged with the unique identification number of SR#1 and serial #; (Sample Receiving #1), the date calibrated and the correction factor. This information must also be recorded in a bound notebook. Only this thermometer can be used for recording the temperature of sample coolers upon receipt.

1. To measure the temperature, open the temperature control blank if supplied, point the IR thermometer at the liquid surface, wait 30 seconds for temperature to stabilize. Read the temperature to the nearest 0.1 °C. The corrected value temperature must also be recorded on the chain of custody. (This value will also be recorded into the LIMS at a later point.). All regulatory compliance samples received from North Carolina that do not meet the temperature requirement will be segregated and the client will be notified of the non-compliance. The samples will not be analyzed until we receive client notification to proceed with analyses.

2. If the temperature exceeds 6°C for any sample, the Project Manager or Sample Receiving personnel must contact the client immediately. There may be tighter temperature control limits for specific project requirements. The customer must make the decision to either continue with the analyses or resample. Make sure the client is aware that if the samples are analyzed, the following qualifier is normally included on the final report: "The shipping cooler temperature exceeded 6°C upon receipt to Empirical Laboratories. This may have an impact on the analytical results. The concentration may be considered as

estimated." Not all samples for the project will be flagged, just those samples received above 6°C.

Many times we are not able to get in touch with the client quickly and the best judgment on how to handle the samples must be made after discussion with the Testing Coordinator and/or Laboratory Director or Technical Director. The samples may still need to go through the log in process although it may be eventually determined that the samples will not be analyzed or the samples may temporarily be placed on hold and not logged in. Above all do not allow the samples to set out at room temperature for an extended period of time while waiting for a decision. **A CAR outlining the problem and all correspondence must be completed.**

The only exceptions to the 6°C rule are:

a. Water samples for all Metals, (except Chrome 6+ and mercury) that have been preserved with HNO_3 to a pH of ≤ 2. *Keep in mind that non-aqueous sample for Metals must be cooled.*

b. Samples for Fluoride, Chloride and Bromide.

c. Waste/Product samples for all parameters.

d. Samples generated in the Aquatic Toxicology laboratories and brought directly to Sample Receiving after they are collected. Sample receiving personnel should place these in cold storage as soon as possible.

e. Samples collected locally by Empirical Laboratories personnel or local customers that hand deliver their samples. In some instances these samples may not have had time to cool down; however, these samples should have been placed on ice in an attempt to cool them to the proper temperature. This exception is only applicable if the samples were collected the same day as the laboratory receives them. It should be noted if samples are "Received On Ice" (ROI).

f. Samples that are received on ice and it is evident that the client made a good faith attempt to properly cool the samples.

F. If several coolers are received at once, they must be inspected to determine the order in which the samples should be unpacked and logged in. The following priorities should be given:

1. Any analyses, which have a 24-72 hour holding, time. It is the log-in person's responsibility to notify the department manager or section group leader of such samples via e-mail and verbally.

2. Any sample which has almost exceeded its' holding time. (Especially watch for this with waters organic extractions, Solids and Sulfides, all of which have only 7 days). A list of parameters and holding times is posted in the log-in room.

   a. If a sample is received already out of holding time, the project manager must be contacted. The sample can be analyzed at the client's request, but it will be qualified on the final report as being analyzed out of holding time. The project manager must inform you of the client's need.

   b. If a sample is received with limited holding time remaining for any parameter it may be necessary to contact the project manager so that he/she can contact the client. If the sample has to be analyzed on a rush basis to meet the holding time a rush charge may apply. Also it may not be possible to analyze the sample within the holding time due to sample load, etc. A CAR must be completed.

3. Samples requiring rush turnaround.

   a. If sample(s) require 24-hour turnaround they will take first priority. Other rush requests also have high priority.

   b. The Project Manager and/or Section Manager must be contacted for approval concerning any unscheduled rush requests.

G. Unpack all samples from the cooler. If there are any known or suspected hazards this must be done under a hood. All coolers from USACE projects should be unpacked under a hood. It may be necessary to rinse off the outside of the containers in the sink and/or wipe them off with a paper towel.

   1. Visually inspect them for tampering and custody seals (if applicable). Sort and inventory the samples against the chain of custody by arranging them in the same order as they are listed on the chain of custody. Normally samples are assigned log numbers in the same order as they are listed on the chain of custody but for certain projects or situations it is acceptable to arrange them in a manner which will make them easiest to log in.

   2. Check for leakage as this could compromise the sample integrity. If any spillage occurred in the cooler make sure this is noted. Also list all the other samples in the cooler as cross contamination could occur. A CAR must be completed and the Project Manager and/or the customer may need to be notified in these situations. It may be necessary to resample.

H. Check the chain of custody information against the information recorded on the containers. If these do not agree, contact appropriate person (s) - Project Manager, sampler, client, etc. All problems must be documented with a CAR.

1. If major changes are made on the chain of custody received from an engineering job, then the PE should submit written confirmation of these changes or make the corrections and initial them directly on the chain of custody.

2. Any error found on the chain of custody must be marked through with one line, initialed and dated and the correction written in.

I. Note any unusual requests, methodology, hazards (known or suspected) to the Project Manager and/or Laboratory Section Manager or analysts before the samples are actually logged in. Keep notes of any problems (improper containers, preservatives, temperature, or descriptions, etc.) A CAR must be completed and the analyst or manager should be notified immediately. If ever in doubt, fill one out!

II. Sample Log In

A. After samples have been unpacked, sorted and reviewed, they are then ready to be assigned log numbers and continue through the log in process. Make sure that the parameters for the samples are clearly marked on the chain of custody. If we prepared the sample kits there should be a sample kit work order form. Contact the Project Manager if there are any questions, problems, etc.

B. Assign a work-order and sample number to each individual sample and record it on each sample container and the chain of custody.

1. All containers with the same description must have the same sample number even if they have different preservatives and require different tests. However, each different fraction (bottle type and/or preservative) should be designated with a letter (A, B, C, etc.)

2. Grab and composite samples from the same sample location must be considered as separate samples. It may be necessary to use "grab" or "composite" as part of the sample description to distinguish between the samples. Only assign different log numbers to them if the parameters are clearly marked as grab and as composite. Do not assume that VOC must be analyzed from grab samples so therefore the client must have taken a grab sample.

3. Sample numbers must begin with 001 at the beginning of each year (e.g. 0101001).

C. Check the following items and record this information on the cooler receipt form to further ensure sample integrity. A CAR must be completed if any of the following requirements are not met and it may be necessary to contact the client. We can perform the analyses in most cases and will do so with the client's approval, however the results may be qualified in some manner on the final report.

Preserving sample integrity throughout the log in procedure must be one of our section's top priorities. This includes not only ensuring that the proper chemical preservatives have been added but also that the samples are received and maintained at the proper temperature. ***When samples are unpacked they must be placed in cold storage within two hours even if they have not been through the entire log in procedure***. All samples for NPDES compliance monitoring from North Carolina will be stored at a temperature range of 1.0 to 4.4°C. All other NPDES samples will be stored at 4.0 ± 2.0°C. On the days we receive a large volume of samples, or are short handed, etc., we may not be able to completely log in all samples until late in the day or even the next day. Samples should not set out at room temperature if there is a delay. The samples must temporarily be placed in cold storage until you are able to complete the log in procedure. This should also be done when we take lunch breaks.

[Make sure the VOC containers are not temporarily stored in a non designated VOC only storage area.]

1. Determine if the samples were received at the proper temperature. (See section IC)

2. The sample descriptions on the bottle should match those on the chain of custody. (See section 1H)

3. Check to determine if the proper chemical preservatives were added to adjust the sample to the correct pH. All regulatory compliance samples received from North Carolina that do not meet the preservation requirement will be segregated and the client will be notified of non-compliance. The samples will not be analyzed until notification to proceed with analyses is received from the client. A list of parameters and the required chemical preservatives is posted in the log-in room. The verification of this preservation will be recorded on the Cooler Receipt Form for all projects. If Empirical Laboratories prepared and shipped out the sample containers they will have been pre-preserved unless instructed otherwise by the client. Complete traceability of the preservatives used to pre-preserve the sample containers and to preserve samples in the log-in area is required. A bound notebook must be used to trace this information and must include the following: Lot #, Type of preservative, Date Prepped, Amount and Analyst Name. This information must also be labeled on each container, re-pipetter, etc. that the preservative is stored in. Each lot of HNO3 used for Metals preservation must be tested prior to using them for preservation. These analyses are kept on file.

   a. The pH of each container (except VOA vials) which requires pH preservation must be checked. Do not open and check the pH of VOA vials in sample receiving/log-in.

   b. The pH of preserved samples is checked and confirmed using pH narrow range indicator paper. When the client request pH analysis on samples and they must be reported and measured for pH using the narrow range paper, rather then a pH meter, the accuracy of each batch of indicator paper must be calibrated to the nearest tenth versus certified pH buffer and recorded into a bound logbook in accordance with SW846 method 9041A pH Paper method.

c. When taking the pH reading, DO NOT PUT THE pH PAPER DIRECTLY INTO THE SAMPLE CONTAINER. Pour up a small aliquot and dispose of this volume after the pH is taken. For some samples (wastes) the indicator paper may not be accurate due to interferences. The observation of the appropriate color change is a strong indication that no interferences have occurred. If it appears as if there is interference, the pH must be measured using the pH meter. [See SOP ATSD-187 pH, Electrometric.]

4. The following guidelines must be followed to check pH preservation:

   a. Water samples for Cyanide analyses must be preserved to a pH of >12 with NaOH upon collection. If the pH of these samples is between 11.0-12.0 upon receipt, and the samples are at the proper temperature and not over 48 hours old it will not be necessary to complete a CAR, however the sample should be adjusted to $\geq$12.0 unless project/client specific requirements are to contact the client first.

   b. Water samples for Metals analyses must be preserved to a pH of $\leq$2.0 with HNO_3 upon collection. If the pH of these samples is between 2.0-.3.0 upon receipt, and the samples are not over 48 hours old it will not be necessary to complete a CAR, however the sample should be adjusted to $\leq$2.0. unless project/client specific requirements are to contact the client first.

   c. Samples requiring analyses which are preserved with H_2SO_4 (i.e., Nitrogen compounds, Total Phenolics, Oil and Grease, Total Phosphorus, etc.) can be accepted up to a pH of 2.5 without a CAR, however the sample should be adjusted $\leq$2.0 unless project/client specific requirements are to contact the client first. Samples for sulfide analysis must have a pH >9.

   d. If a sample is not properly preserved, log-in personnel must either do the following:

      - To meet project specific requirements, including all USACE projects, the client must be notified before preserving or adding additional preservative to the sample unless otherwise instructed. If the client instructs us to add chemical preservatives to a sample, complete traceability of the preservatives used is required (See section IIC, #3). A CAR must be completed.

      - For other projects it may be acceptable to preserve the sample accordingly before the sample is placed in storage. Complete traceability of the preservatives used is required (See section IIC, #3). A CAR outlining the project and the steps taken must be completed.

      - All metals samples preserved upon receipt must be held 24 hours before proceeding with analysis. These samples must be CAR generated and the client notified to see if the lab is to proceed with analysis.

e. In some instances it may not be possible to adjust the sample to the proper pH due to matrix problems which cause excessive foaming or require an unusually large amount of acid. Do not continue to add acid if a few mL's of acid does not lower the pH. Notify the Project Manager, Metals Manager and/or analyst. They will make the decision if the sample will be diluted, not analyzed, etc. A CAR must be completed in these situations. Make sure you note on the container and in the LIMS notes that the sample is not at the proper pH as well as any useful information (i.e., foaming, strong odor, etc.).

f. A CAR may not be required for samples generated in the Aquatic Toxicology Laboratories and brought directly to Sample Receiving after they are collected but before they are preserved. Log-in personnel must preserve the samples accordingly before they are placed in storage. Complete traceability of the preservatives used is required (See section IIC, #3). A CAR outlining the project and the steps taken must be completed.

5. Check to make sure samples are in proper containers and that there is adequate volume for all the parameters requested and no leakage.

6. If VOA vials are present, each vial must be inverted and checked for head space. "Pea-sized" bubbles (i.e. bubbles not exceeding 1/4 inch or 6 mm in diameter) are acceptable and should be noted, however, a CAR is not required. Large bubbles or head space is not acceptable and a CAR must be completed. If this occurs, the client must be contacted. The samples can be analyzed with their approval, however the report will be qualified and the data may be questionable. All VOA vials will be preserved with $Na_2S_2O_3$ (0.2g) when chlorine is known to be, or suspected to be present.

7. All pesticide samples to be analyzed by method 608 will be checked by the sample receiving personnel for the correct pH range of 5.0 to 9.0. The pH of the sample(s) will be communicated via E-mail to the Section Manager and appropriate analyst.

8. All chlorinated effluent samples received for Cyanide must be checked for residual chlorine. The one liter sample container should initially contain 1 to 2g/L of Ascorbic Acid. Potassium Iodide starch indicator paper will be used for detecting the presence of residual chlorine. DO NOT PUT THE TEST PAPER DIRECTLY INTO THE SAMPLE CONTAINER. Pour up a small aliquot, neutralize, test and dispose of this volume after the sample is checked. If the test paper turns blue, the sample must be treated for residual chlorine. Add Ascorbic Acid, approximately 0.6g at a time and recheck the sample until there is no residual chlorine present. If the sample required this treatment this information must be included in the LIMS notes. This must be done by log-in personnel before leaving the receiving area. It may be necessary to notify the Inorganic Manager and/or analyst.

9. Be aware of holding time requirements. (See section 1D)

D. Once sample containers have been numbered, they must be checked by another laboratory individual to ensure that the log number on the container matches the log number and sample ID on the Chain of Custody. A ***Sample Receiving Custody and Disposal Form [Attachment VII]*** must be completed each day. Samples should not leave the log-in area until this has been completed. *[see IIC; it may be necessary to temporarily store samples in cold storage until the samples can be second checked, the amount of time that the samples are at room temperature must be minimized as much as possible.]* The original is to remain in Sample Receiving until the samples are disposed of. Once the document is complete, the original will be kept on file. The following information must be logged onto this form:

   1. Client and Log #s
   2. Date/Time Unpacked
   3. Logged In/Numbered By (Initials)
   4. 2nd Checked By (Initials)
   5. Date/Time Placed in Cold Storage
   6. Storage Area (Walk In, VOC Cooler, Quarantined Soils, Quarantined-VOC, Other)
   7. Disposed of By/Date
   8. Method of Disposal

E. Notify the proper analyst if samples have been logged in for analyses which have a 24-48 hour holding time or if a 1-2 day turnaround has been requested. The log number and description on sample (s) must be second checked before it is released to the analyst. (The analyst can second check the sample, but must initial the custody form.)

III. Sample Storage

A. After samples have been correctly logged in they are then transferred to one of the following cold storage areas and arranged in numerical order by the assigned log in/LIMS sample number. ***Note that aqueous VOC samples must be segregated from all other samples.***

   1. The Hobart refrigerator in the MS Lab: All aqueous VOC's must be stored in this refrigerator. Storage blanks consisting of organic free water from the laboratory may be required for specific projects. These will be analyzed for VOCs only. ***Storage blanks are required for all DOD projects.***

   2. Walk In Refrigerator: All aqueous samples for all analyses must be stored in this refrigerator.

3. Soil Walk-In Refrigerator: All quarantined and non-quarantined soil samples for all analyses must be stored in this refrigerator.

B. Quarantined soils are those quarantined by the US Department of Agriculture. These soil samples must be segregated from other soil samples during storage. A separate disposal log must be maintained for these soils including the location, date and quantity of the soil received and processed. Soil residues from quarantined samples must be treated according to regulations after testing (see Sample Disposal SOP). Quarantined soils are defined as:

1. Soil taken from much of the southeastern US and parts of New York and Maryland at a depth of three feet or less. *Soils from three feet or more are not regulated provided they are stored separately.* A map of the regulated areas in the United States entitled ***Soil Movement Regulations [Attachment VIII]*** is posted in the log-in room.

2. All soils taken from foreign sources, US. Territories and Hawaii.

NOTE: All soils are treated as quarantined soils and are disposed of in accordance with USDA regulations. Above for information purposes only.

C. All samples must be stored in one of the three refrigerators detailed above with the following exceptions:

1. Matrices that may be adversely affected by the cold temperature. (e.g. surfactant samples, multi-phase samples)

2. Highly contaminated waste or product type samples that could jeopardize the integrity of other samples in the walk in cooler. Often these can be stored at room temperature. If these require refrigeration see the Project Manager for other options.

D. The temperature of each sample refrigerator must be monitored and recorded each day by Wet Chem personnel by the following method. A Mercury thermometer or digital min/max thermometer with 1° increments must be used. Each thermometer must be calibrated against a NIST certified thermometer once a year **(digital thermometers quarterly)** and this information recorded in a bound notebook. The Certificate of Calibration for the NIST thermometer is kept on file at the QAO's desk. The thermometers must be tagged with a unique identification, the date calibrated and the correction factor.

The tolerance range for all refrigerators is 1 to 6°C. This range and the range using the corrected reading must be posted on the outside of each cooler. If the temperature exceeds this range, corrective action measures must be put in place immediately. A CAR must be completed specifically noting the date and time the problem was discovered. The Project Manager, Laboratory Director and Technical Director will be notified in order to assess the situation. It may be necessary to put a service call in to the refrigeration repair service.

E. All personnel removing samples from any refrigerator must sign them in and out. This is done by completing the ***Sample Custody Form [Attachment IX]*** which is attached to the door of each refrigerator. These completed forms are kept on file [see section III, #4F]

F. The water walk in refrigerator in the sample room is the largest refrigerator and stores a large majority of the samples. A back up compressor is hooked into the system and scheduled to automatically come on if the main compressor fails. There is a digital min/max thermometer, which monitors the temperature 7 days a week. This thermometer will be calibrated quarterly against the NIST thermometer.

G. As stated above the temperatures for all refrigerators that samples are stored are checked each day Monday-Friday and monitored seven days a week with min/max thermometers. Pay close attention to these readings and watch for signs of possible problems.

H. A temperature maintenance record book is kept for each refrigerator.

I. Samples must be held for a minimum of 30 days after the final report unless specified otherwise. For USACE projects, samples must be held for a minimum of 60 days after the final report unless otherwise specified. See SOP ATSD 405 entitled Analytical Laboratory Waste Disposal SOP for guidance on disposal of samples.

IV. Laboratory Information Management System (LIMS)

A. Log the sample information into the LIMS for each sample. Every attempt should be made to get every sample logged into the LIMS by the end of the day. All information entered should be clearly stated and recorded on the COC provided. After opening the main menu of the LIMS, select the 'Work Orders' tab from the 'Sample Control' drop down menu. Now click on the 'Add' button to create a new Work Order. You will see the following:

1. ***Client***:

Select the client I.D. by clicking on the pull-down and choosing from the client list. This list is in alphabetical order. If the desired client is not on the list, a new client must be created by the project manager or I.T. director.

2. ***Projects***:

Click on 'Projects' and choose the project I.D. The projects will be client specific. After the project is chosen the "project information" areas should fill in. The 'Project Name,' 'Project Number,' 'TAT,' 'Client Project Manager,' 'Lab Project Manager,' and 'Comments' information should also appear. If there are no applicable project choices, a project must be created by the project manager or I.T. director. There are two types of projects:

a. Internal -- Empirical Laboratories projects;

b. External -- direct laboratory clients.

3. ***Comments:***

This area is to be used to note any information from the project manager for all work orders of this project. It can also be used to list any work order specific notes; this includes but is not limited to information concerning rush turnaround, deliverables or other QC requirements, analyte concentrations, safety issues, quarantined soils, CAR #s, preservation or matrix problems, etc.

4. ***Received By:***

Enter the name of the person who received the samples.

5. ***Logged In By:***

Enter the name of the person who logged in the samples.

6. ***Received:***

Enter the date and time received separated by a space and using military time.
Example: *08/02/2008 08:30*

7. ***Project/Package Date Due:***

After the date and time received have been entered, the date due for both of these fields will be calculated. If this information is not correct or needs to be amended later, check with the project manager before doing so.

8. ***Shipping Containers:***

Click on the 'Coolers' button and enter the temperature and condition upon receipt. If more than one cooler was received, each cooler must be assigned a different name. For example, if these came in by dedicated courier, enter the last four numbers of the Tracking Number as the name. After all of a cooler's information has been entered (received on ice, where custody seals present, preservation confirmed, COC/container labels agree, sample containers in-tact) click the 'Save' button. If more than one cooler was received, click the 'Add' button and repeat the process above, then click 'Done' after all the coolers' info has been saved.

9. ***COC Number:***

If an identifiable COC number is listed, record that ID here.

10. ***Shipped By:***

Enter the courier used to deliver the samples. If the samples were picked up by a lab employee or dropped of by the client/representative, enter 'Hand-Delivered.'

After these items have been completed, click 'Save,' then the 'Samples' button to continue. To begin entering information for a sample, click the 'Add' button on the bottom of the Samples screen.

11. ***Sample Name:***

    a. Only abbreviate if description is too long for the spaces allotted in the LIMS. This information should come directly from the chain of custody. The sample ID entered into the LIMS will be the sample ID on the final report.

    b. If no sample ID is provided, or is indistinguishable from other samples listed, contact the project manager to ascertain distinction in the samples. Include date as part of the description if this is the only way to differentiate the samples.

    c. When logging in trip blanks that do not have an ID assigned by the client, list them as "Trip Blank # ______". This information should be on the containers. A log book must be kept in the sample kit room which lists all trip blanks and the date they were filled. This will ensure consistency with the descriptions for trip blanks. Make sure you record the trip blank on the chain of custody if it is not listed.

12. ***Collection Date:***

Enter the date and time the sample was collected. You must use military time and separate by a space. Often the time collected is not given. Although this is a sampling requirement, this information may not be crucial unless a parameter with a short holding time or a data deliverables package is required. In the event that a sample collection time is not listed on the COC or the sample container, a default time of 00:00 can be used temporarily until client verification. Once verified, then the correct sample collection time must be input into LIMS. If the COC and sample containers do not list a collection date and time, a CAR must be generated. All attempts should be made to get all our clients to supply this information.

13. ***Lab/Report Matrix:***

Click on pull down and select matrix. Many times it is difficult to discern the matrix if it is not specified on the COC, and log-in personnel must use their best judgment with regard to analytes/methods requested. Keep in mind that the detection limits and units on the LIMS reports are linked to the matrix. In some cases it may be necessary to ask the Section Managers about the matrix selection. Log-in may do a dilution test to distinguish water samples from oil samples if the COC does not clarify a sample matrix if need be.

14. ***Sample Type:***

This is used to differentiate between special types of samples (i.e. Field Duplicates, Equipment Blanks, Trip Blanks, etc.). If there is no definite way to determine that a sample should be classified as something else, then "SAMP-Client Sample" will be selected as the sample type. Do not list a sample as anything other than a Client Sample unless noted on the COC of are instructed by the client to do so.

15. ***Container:***

Click on the drop down list and select the appropriate bottle type. If multiple bottles are received for the same sample, then move down to the next line and select all other containers as required. Repeat this process until all containers for the sample are listed. As each container is entered, an individual number is assigned to it by the LIMS system. This number is also listed on the container labels that are printed from the LIMS, and is placed on the corresponding bottle for container tracking purposes.

16. ***pH*** (Container Preservative)***:***

Use this to document the pH check information taken during sample unpacking. If no preservative was used, then nothing is required in this field.

17. ***Comments:***

Enter any information that is applicable at the sample level.

18. ***Field Analysis:***

Click on field analysis tab and enter field information when provided.

19. ***Work Analyses:***

Select all parameters requested for the sample from this list.

a. If the required test code is not listed, and the sample matrix is not a contributing factor, click the Work Analyses tab to open the All Analyses list. When selecting from this expanded list, be careful to select the proper method as all methods available for the current matrix will be selectable.

b. If any analyses are selected from the All Analyses list, the Project Manager in charge should be notified so that the correctness of methods and pricing can be checked and updated as needed.

c. All preparation codes for analytes are entered and stored by the system independently of the test codes selected, except in the cases of Dry Weight analysis, and TCLP/SPLP preparation (tumbling). In the case of the TCLP/SPLP prep codes, these are entered alongside the other required analyses automatically by the LIMS when a TCLP/SPLP analyte is selected. As for Dry Weight, it is required for all solids testing except in the cases of TCLP/SPLP analysis, Explosives only analysis, and/or any pure product/non-soil based sample when specified by the client.

20. ***Analyses Comments:***

These comments should be used for any notes that only apply to that particular test code.

21. ***RTAT:***

If the Rush Turn-Around Time for this sample is known at the time of log-in, this information should be updated here.

22. ***Save:***

Once all applicable information is entered for a sample, click the save button. At this time the LIMS applies the Laboratory Sample ID to the sample. This is a four part ID code composed of the following:

a. A 2-digit numeral of the year. Example (**08**11248-06)

b. A 2-digit numeral of the month. Example (08**11**248-06)

c. A 3-digit numeral of the work order number. This number reset to 001 at the beginning of each month. Example (0811**248**-06)

d. A 2-digit numeral of the sample number separated by a dash. Example (0811248-**06**). This number is different for each sample in a work order, and a single work order cannot contain more than 99 samples. If more sample numbers are needed, a new work order number will have to be assigned to the applicable set of sample.

23. ***Add/Edit/Copy:***

Use these selections to add more samples to the work order, or to change existing information prior to label printing.

Once all the tests have been selected and all samples have been added in the work order, a work order summary and all container labels are printed. Labels are checked for accuracy against the containers while being labeled. At this point log-in of this group of samples is complete.

B. After log-in of a work order is complete, the COC can then be scanned into the system, attached to the work order on the Work Order screen, and the work order can be updated to Available status so as to be seen by the analysts.

V. Daily Follow Up for Sample Receiving/Log In

A. Wipe out the inside of coolers and return all Empirical Laboratories coolers to the sample kit room. Discard any coolers that are cracked, broken or filthy.

B. If any samples were received for RUSH turnaround, then a ***RUSH SHEET [Attachment XII]*** must be completed and distributed to all laboratory personnel via e-mail. If ever in doubt of which analysts should be notified, pass them out to everyone. Always give copies to the Laboratory Director, Administrative Assistant and Section Managers. It may be necessary to send out two RUSH sheets per day (one around mid-day and the other at the end of the day).

C. Complete any required CARs.

D. At the end of the day organize all paperwork received and generated for the day. The following should be given to the Project Manager (section supervisor):

1. The original chains of custody and yellow original or copy of each. The CRF will accompany the COC for the project.

2. Any information (letters, regulatory limits, etc.) from a client which was received with any samples.

3. All CARs.

4. LIMS sample receiving logs.

5. Copies of any RUSH sheets which have been distributed

6. Sample Receiving Custody and Disposal Form.

7. Cooler receipt form.

E. All the above information from the day will be reviewed as soon as possible.

1. All LIMS logs must be 2nd checked by a different person than the person entering the information into the LIMS. Each set of logs must be initialed dated by the person 2nd checking. These will be kept on file at the Project Manager desk.

2. If any corrections or changes are required, all laboratory personnel will be notified by distributing a ***Sample Log Change Form [Attachment XIII]*** through email distribution. A ***Sample Log Change Form*** by the project manager will also be sent out if a client adds or deletes any parameters, changes sample IDs, etc.

F. The Testing Coordinator will distribute the following after they have been through the 2nd QA check:

1. Copies of the LIMS receiving reports to necessary laboratory personnel.

2. Original (white copy) chains of custody are given to the Project Manager. These will be sent with the final report to the client.

3. Finalized/approved CARs must be sent to the:

a. Organic Manager

b. Inorganic Manager

c. Laboratory Manager

d. Laboratory Director {optional}

e. Quality Assurance Officer

f. Administrative Assistant

g. Client {optional}

4. Copies of any project/sample specific information to the Section Manager and analysts.

G. Information will be filed as follows:

1. Chains of custody:

a. Original (white copy) is returned to the customer with the final report along with the CRF.

b. Pink copies should be retained by the sampler.

2. CARs

a. CARs can be found at V:\LAB\log-in\login (year)\logcar (year).

3. Sample Change Forms and RUSH Sheets

a. Sample Change Forms are distributed by email.

b. RUSH Sheets are found at V:\LAB\login\Rushsheets

4. At the end of each year, files for that year are boxed and archived. Make sure files are labeled properly and place them in banker's boxes. Complete a storage box file form with as much detailed information as possible. The Laboratory Administrative Assistant will label and number the boxes and incorporate the storage boxes into the laboratory file archive system. Boxes containing files from Sample Receiving are kept on site for 1-2 years and then may be moved to off site storage upon release from the Project Manager.

VI. Miscellaneous

A. All projects which require deliverables or other QC requirements should be listed in the notes section of the LIMS.

B. If samples are received from a new client or a new job number that is not in the LIMS, a new client code must be set up. This information should be on the chain of custody or it may be necessary to contact the customer if the information is incomplete.

C. Samples from the Aquatic Toxicity Laboratory (ATL) are logged into the LIMS for billing and long-term tracking purposes. The receiving information and proper assignment of tests are reviewed by the ATL Manager. The samples are then logged in by ATL personnel.

D. A flow chart outlining sample receiving and the flow of data, reporting and invoicing is attached as ***Attachment XIV.***

E. A ***Telephone Conversation Log [Attachment XV]*** may be required to document information and may be attached to or used as a CAR.

F. All log books used in the Sample Receiving and Sample Storage Areas are numbered. The following log books are presently maintained. All log books must be "Z"ed out. The Testing Coordinator will review the log books each week to check for completeness.

Log Book ID	**Log Book Description**
LI014	Trip Blank Prep Log Book
LI009	Tracking of VOC Trip Blanks Shipped
LI011	Quarantined Soil Treatment Log Book
LI012	Acid Neutralization Log Book
LI013	Sample Receiving and Disposal Log Book
LI010	Kit Room Preservation Preparation Log Book

Attachments to SOP 404

II	Chain of Custody Record
III	Corrective Action Report for Sample Receiving/Log In
IV	Cooler Receipt Form
V	List of Short Holding Time (Immediate-72 hrs.) Parameters
VII	Sample Receiving Custody and Disposal Form
VIII	Map of Quarantined Soil Areas in the US.
IX	Laboratory Sample Custody Form for Walk In Refrigerator
X	Container Codes for the LIMS
XI	Routine NPDES Clients
XII	RUSH Sheet
XIII	Sample Log Change Form (Green Sheet)
XIV	Flow Chart, Laboratory Sample Tracking System

[Attachments I and VI were removed during the editing process and not added to the SOP.]

EMPIRICAL LABORATORIES, LLC - CHAIN OF CUSTODY RECORD

SHIP TO: 227 French Landing Drive, Suite 550 ♦ Nashville, TN 37228 ♦ 615-345-1115 ♦ (fax) 615-846-5426

Send Results to:	Send Invoice to:	Analysis Requirements:	Lab Use Only:
Name ____ Company ____ Address ____ City ____ State, Zip ____ Phone ____ Fax ____ E-mail ____	Name ____ Company ____ Address ____ City ____ State, Zip ____ Phone ____ Fax ____ E-mail ____		VOA Headspace Y N NA Field Filtered Y N NA Correct Containers Y N NA Discrepancies Y N NA Cust. Seals Intact Y N NA Containers Intact Y N NA Airbill #: ____ CAR #: ____
Project No./Name:	Sampler's (Signature):		

Lab Use Only Lab #	Date/Time Sampled	Sample Description	Sample Matrix													Comments	No. of Bottles	*Lab Use Only* Containers/Pres.

Sample Kit Prep'd by: (Signature)	Date/Time	Received By: (Signature)	REMARKS:	Details:
Relinquished by: (Signature)	Date/Time	Received By: (Signature)		Page ____ of ____ Cooler No. ____ of ____
Relinquished by: (Signature)	Date/Time	Received By: (Signature)		Date Shipped ____
Received for Laboratory by: (Signature)	Date/Time	*Temperature*		Shipped By ____ Turnaround ____

Distribution: Original and yellow copies accompany sample shipment to laboratory; Pink retained by samplers.

Attachment III

CAR 09-

EMPIRICAL LABORATORIES

CORRECTIVE ACTION REPORT FOR SAMPLE RECEIVING/LOG-IN

Date Completed:

Form Completed By:

Date Samples Received:

Parameter(s):

Client/Job #:

Samples:

Problem(s):

Action Taken:

Action Taken to Prevent Reoccurrence of this Problem:

Approval of Section Leader: ______________________________

Distributed to:

EMPIRICAL LABORATORIES
COOLER RECEIPT FORM

LIMS Number: ______________________ Number of Coolers: ________ of __________

Client: ______________________ Project: ______________________

Date/Time Received: Date cooler(s) opened:

Opened By (print): (signature): ______________________

Circle response below as appropriate

1. How did the samples arrive?: FedEx UPS DHL Hand Delivered

EL Courier Other: ______________________

If applicable, enter airbill number here: ______________________

2. Were custody seals on outside of cooler(s)? .... Yes No

How many: __________ Seal date: ______________ Seal Initials: __________

3. Were custody seals unbroken and intact at the date and time of arrival? .... Yes No N/A
4. Were custody papers sealed in a plastic bag included in the sample cooler? .... Yes No N/A
5. Were custody papers filled out properly (ink, signed, etc.)? .... Yes No N/A
6. Did you sign custody papers in the appropriate place for acceptance? .... Yes No N/A
7. Was project identifiable from custody papers? .... Yes No N/A
8. If required, was enough ice present in the cooler(s)? .... Yes No N/A

Type of Coolant: WET DRY BLUE NONE Temperature of Samples upon Receipt: __________

Dates samples were logged-in:

9. Initial this form to acknowledge login of sample(s): (Name): **WILLIAM SCHWAB** (Initial):__________

10. Were all bottle lids intact and sealed tightly? .... Yes No N/A
11. Did all bottles arrive unbroken? .... Yes No N/A
12. Was all required bottle label information complete? .... Yes No N/A
13. Did all bottle labels agree with custody papers? .... Yes No N/A
14. Were correct containers used for the analyses indicated? .... Yes No N/A
15. Were preservative levels correct in all applicable sample containers? .... Yes No N/A
16. Was residual chlorine present in any applicable sample containers? .... Yes No N/A
17. Was sufficient amount of sample sent for the analyses required? .... Yes No N/A
18. Was headspace present in any included VOA vials? .... Yes No N/A

If Non-Conformance issues were present, list by sample ID: ______________________

______________________ CAR#: __________

Short Holding Time Parameters

(Immediate-72 hours)

Parameter	Holding Time
pH	Immediate [a]
Sulfite	Immediate [a]
Temperature	Immediate [a]
Residual Chlorine	Immediate [a]
Coliform (Fecal and Total) RCRA/WW	6 hours
Hexavalent Chromium (Cr +6)	24 hours
Odor	24 hours
Coliform (Fecal and Total) *Drinking Water only*	30 hours
BOD	48 hours
Color	48 hours
Settleable Solids	48 hours
MBAS	48 hours
Orthophosphate	48 hours
Turbidity	48 hours
Nitrite	48 hours
Flashpoint	72 hours [b]

[a] Immediate generally means within 15 minutes of sample collection.

[b] This is an internal holding time. The method does not specify a holding time.

Empirical Laboratories, LLC

SAMPLE RECEIVING CUSTODY AND DISPOSAL FORM

Date Samples Received ____________________

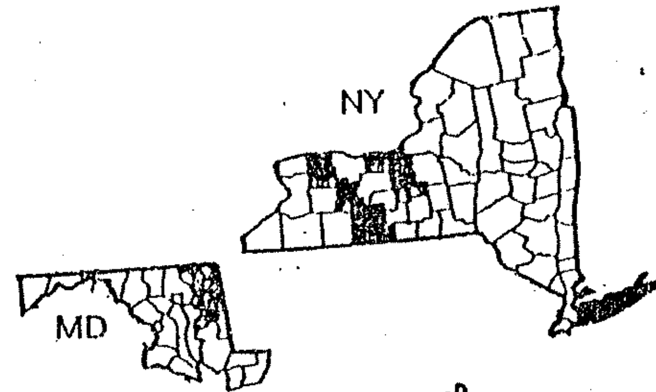
Client	Log #'s	Date Unpacked	Logged In/ Numbered By	2nd Checked By *	Placed in Storage By	Date Placed in Storage	Storage Area (1)	Disposed of By/Date	Method of Disposal

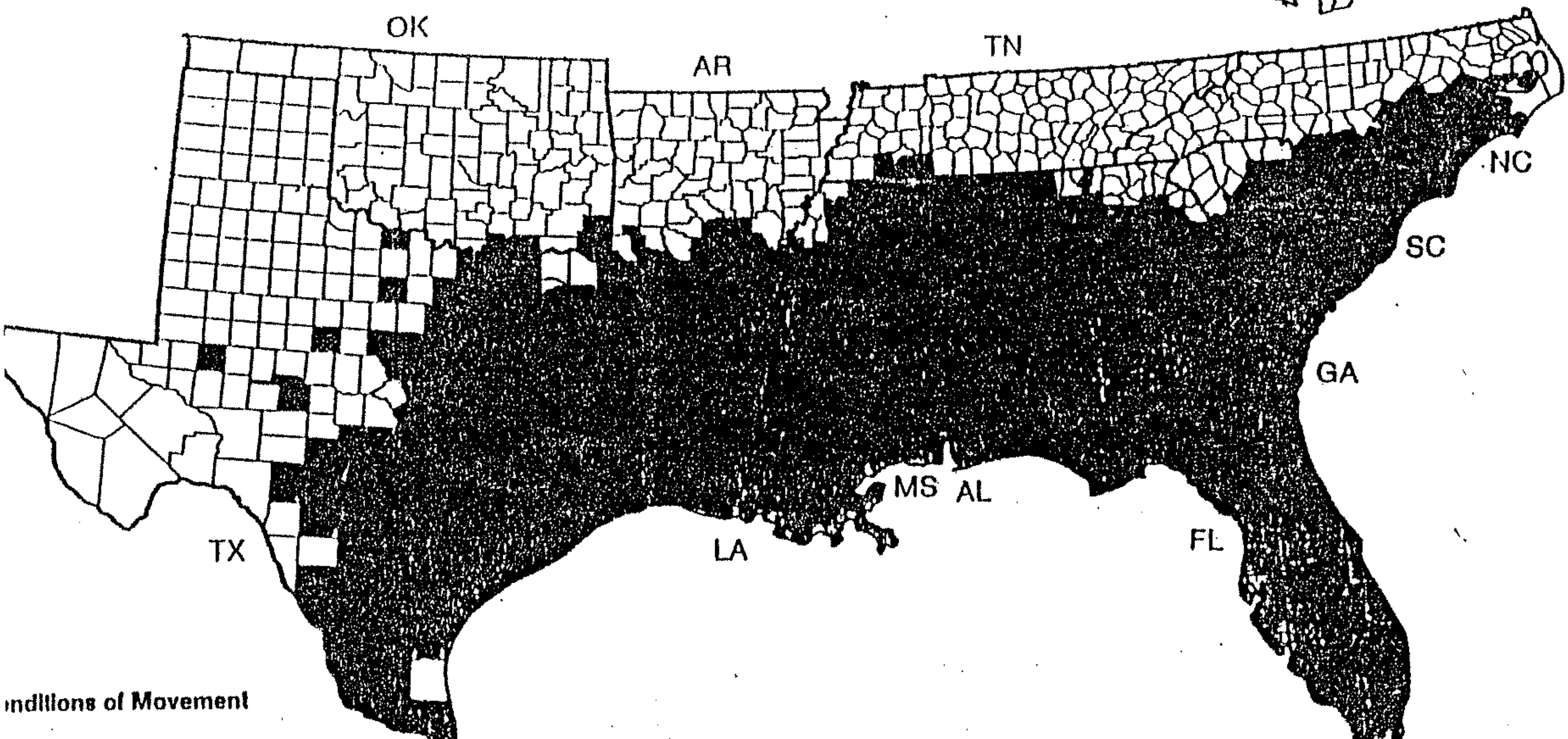
(1) W=Walk in Refrigerator
HV= Hobart Refrigerator, VOC samples only
Q= Refrigerator for Quarantined Soil Samples
QV= Refrigerator for Quarantined Soil, VOC samples only
O=Other (Must Specify)

***= MUST HAVE INITIALS OF THE PERSON WHO SECOND CHECKED AND MUST BE FILLED IN BY THAT PERSON IN INK .**

oil Movement Regulations

(Quarantined Soils by US Dept. of Agriculture)





›nditions of Movement

›strictions are imposed on the move-
›nt of regulated articles from a regulated
›a as follows:
›om red areas into or through white
›eas. Movement within red areas may be
›gulated.

Consult your State or Federal plant protection inspector or your county agent for assistance regarding exact areas under regulation and requirements for moving regulated articles.

Regulated Area

ATTACHMENT VIII

Empirical Laboratories

EMPIRICAL LABORATORIES, LLC
LABORATORY SAMPLE CUSTODY FORM
WALK-IN REFRIGERATOR

Sample Log # (s)	Time/Date/Initials Removed	Time/Date/Initials Returned (Note if all Sample Used)	Notes/ Comments	Task Performed

Preservatives		Types of Container	
NI	*HNO3*	**A**	*1 LITER - PLASTIC*
NF	*HNO3 (Filtered)*	**B**	*500 mL - PLASTIC*
SU	*H2SO4*	**C**	*250 mL - PLASTIC*
SH	*NaOH*	**D**	*120 mL - PLASTIC*
ZN	*ZnAC / NaOH*	**EN**	*ENCORE PAK*
HY	*HCl*	**F**	*1 LITER - GLASS CLEAR WIDE MOUTH*
		G	*1 LITER - GLASS CLEAR BOSTON ROUND*
		H	*1 LITER - GLASS AMBER*
		I	*250 ml. - AMBER*
		J	*VOA VIALS - (40 ml.)*
		K	*500 ml. - (16 oz)*
		L	*250 ml. - (8 oz)*
		M	*125 ml. - (4 oz)*
		N	*60 ml. - (2 oz)*
		O	*OTHER*
		P	*PLASTIC BAG -1 Gallon*

ROUTINE NPDES CLIENTS (Page 1 of 2)

LCAN Ingot and Recycling
moco Oil
rmstrong (Pirelli)
lf Atochem-Carrollton, KY
uburn Hosiery Mill
utostyle

ando Manufacturing
owers Ink
owling Green Municipalities (City of)
P Oil
remner, Inc.
rentwood, City of
rown Printing Central

argill Steel and Wire
larksville Products

. I. Dupont
r and Olsen
mhart Pop Rivets

leet Design
ranklin, City of

atlinburg

Hennessey Co. (Coats)
H.I.S. Laundry
H.K. Bell (City of Hopkinsville Landfill Monthly Monitoring)
Hoover

International Paper
J. S. Technos

Ken Koat
King Industries

annom Tannery
Leonard Plating

al Plate, Inc.
Morflex, Inc.

Nashville Wire
Norandal USA Inc.

Oak Ridge, City of

Plymouth Tube
Prime Colorants

RMI

Shared Hospital Services
Snap On Tools
Springfield, City of
Special Metals
te Industries

Tennessee Dickel Distillery
Tullahoma, City of

UCAR, Clarksville

Valmore Leather

Westvaco (Mayfield Creek Up/Down)
Woodbury

Revised 9/10/96

Date Distributed 1/01/02

RUSH

DATE RECEIVED	CLIENT	LOG # (S)	PARAMETERS	E-MAIL COPY DUE (DATE / TO)	FAX COPY DUE (DATE / TO)	*FINAL COPY DUE (DATE / TO)

*ALL FINAL REPORTS SHOULD BE DELIVERED TO TERESA WILLIAMS OR HERBIE JOHNSON THREE DAYS PRIOR TO FINAL DUE DATE TO CLIENT.

ATTACHMENT XII

96-0001

SAMPLE LOG CHANGE FORM

DATE:

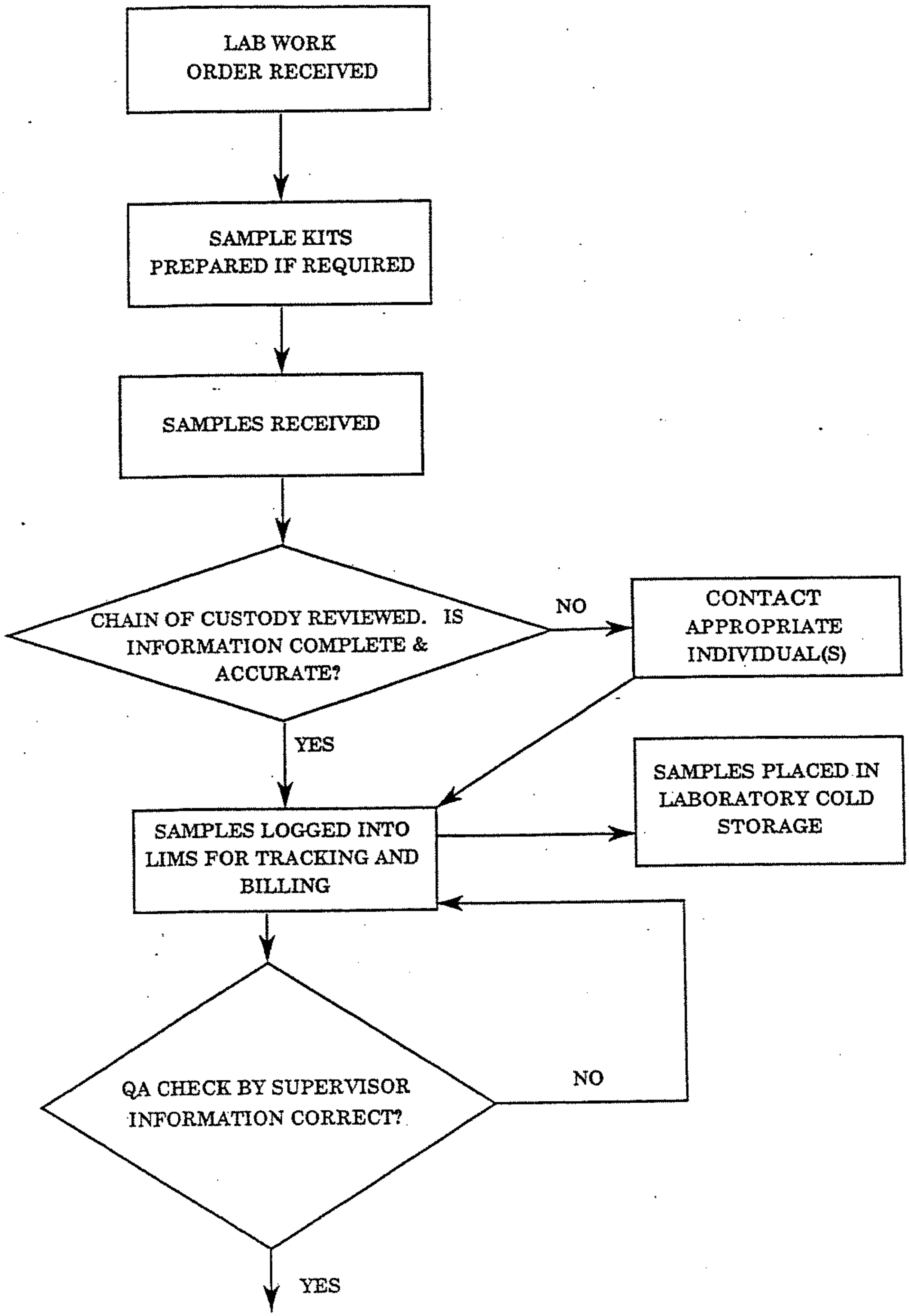
TO:

SAMPLE #(S):

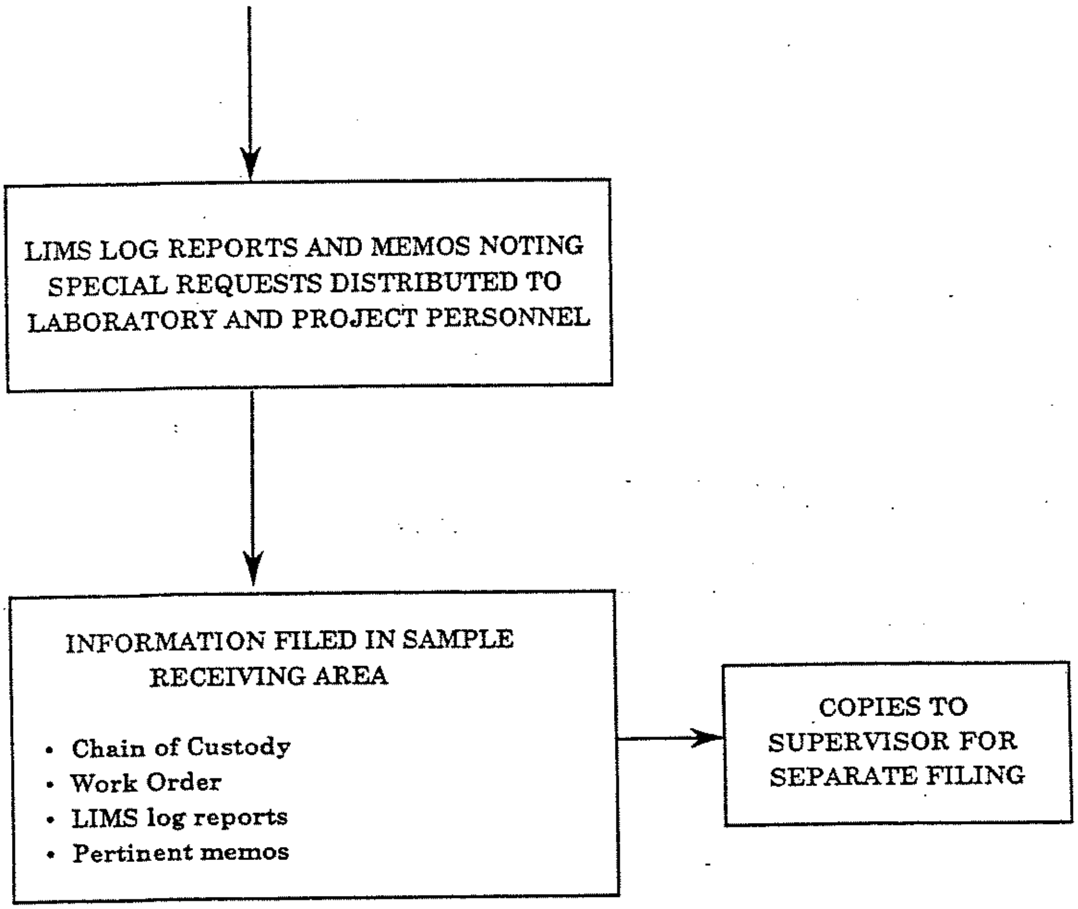
CLIENT:

Changes:

LABORATORY SAMPLE TRACKING SYSTEM
SAMPLE RECEIVING

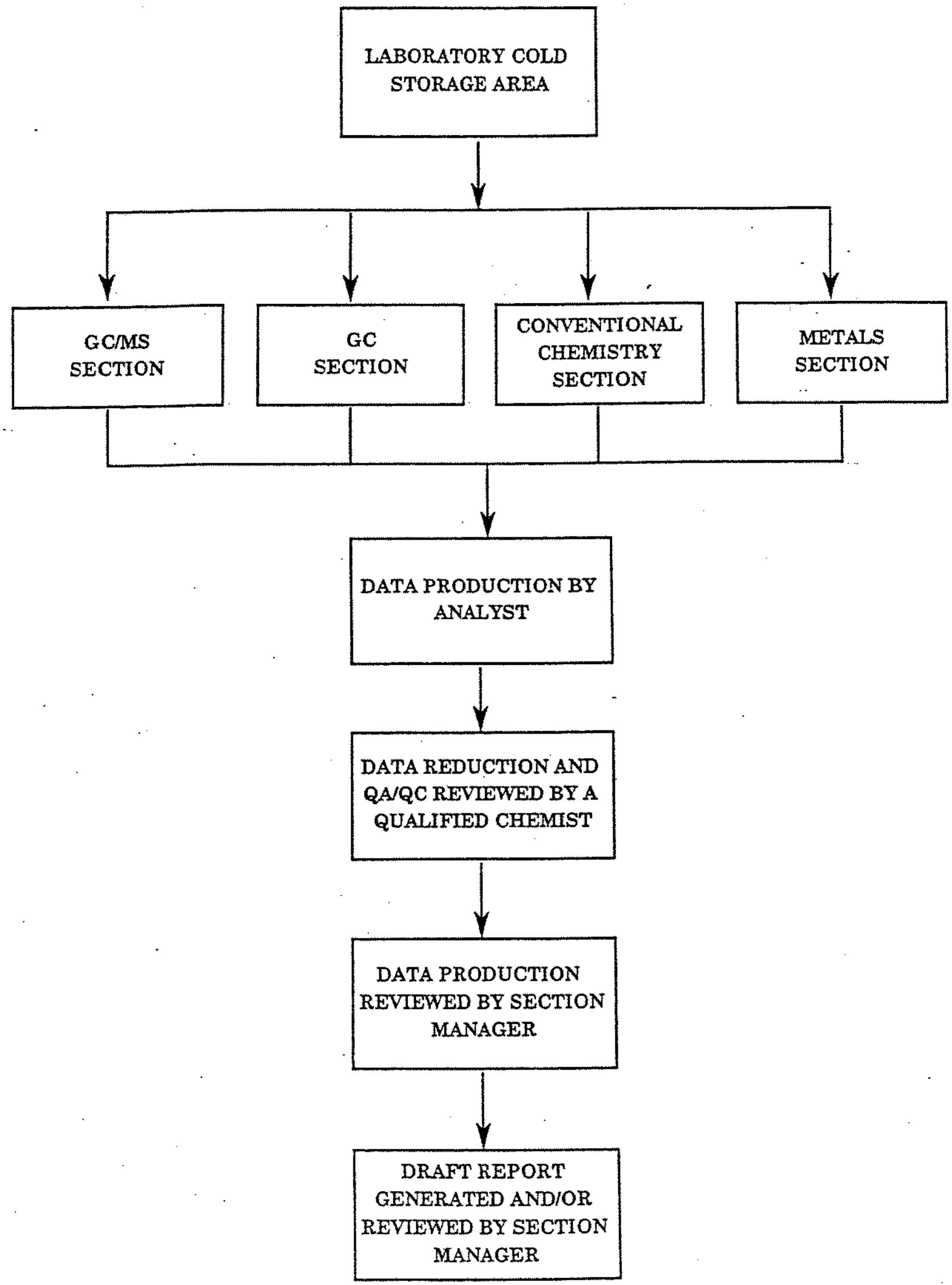


LABORATORY SAMPLE TRACKING SYSTEM
SAMPLE RECEIVING (continued)

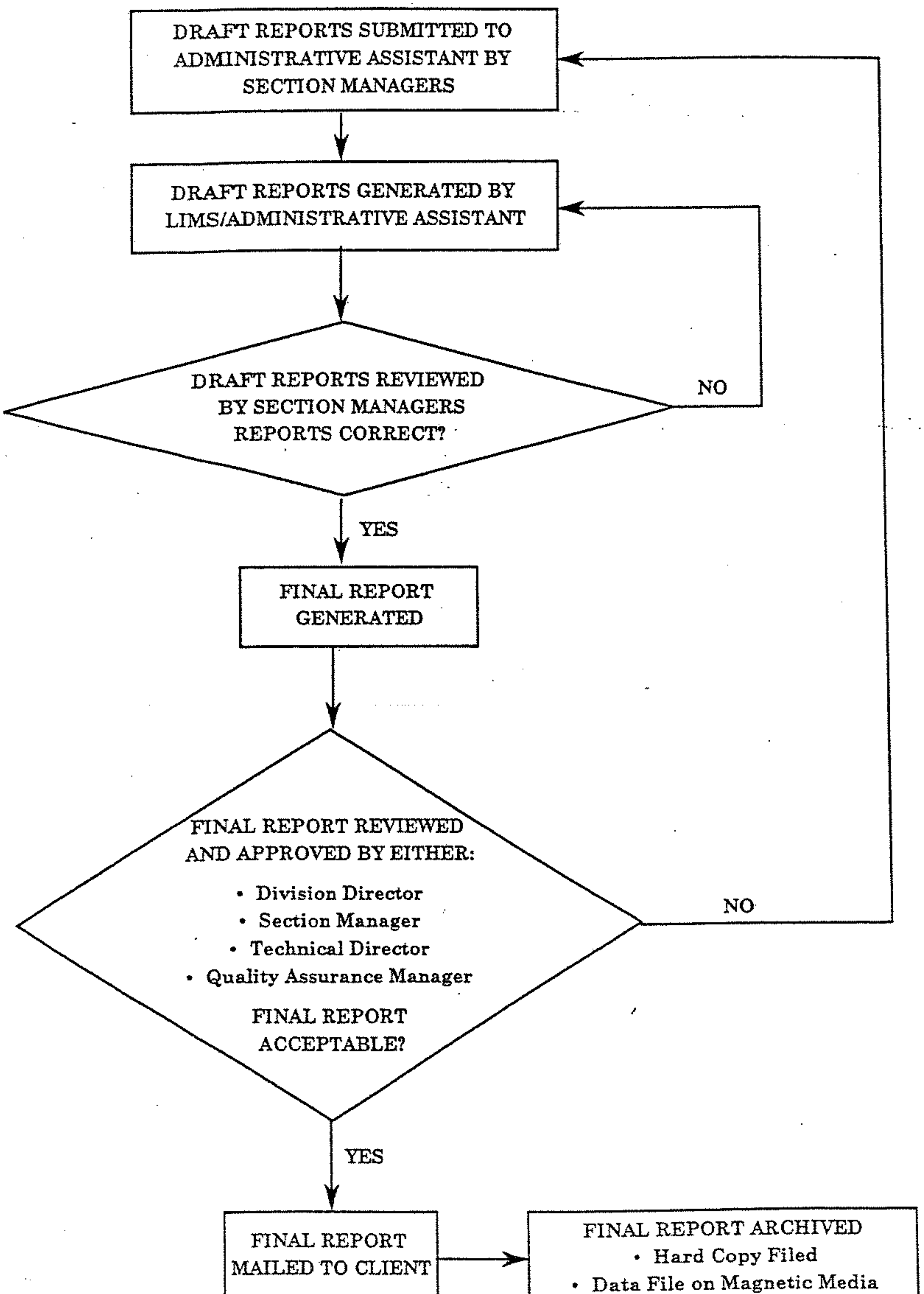


ATTACHMENT XIV (Continued)

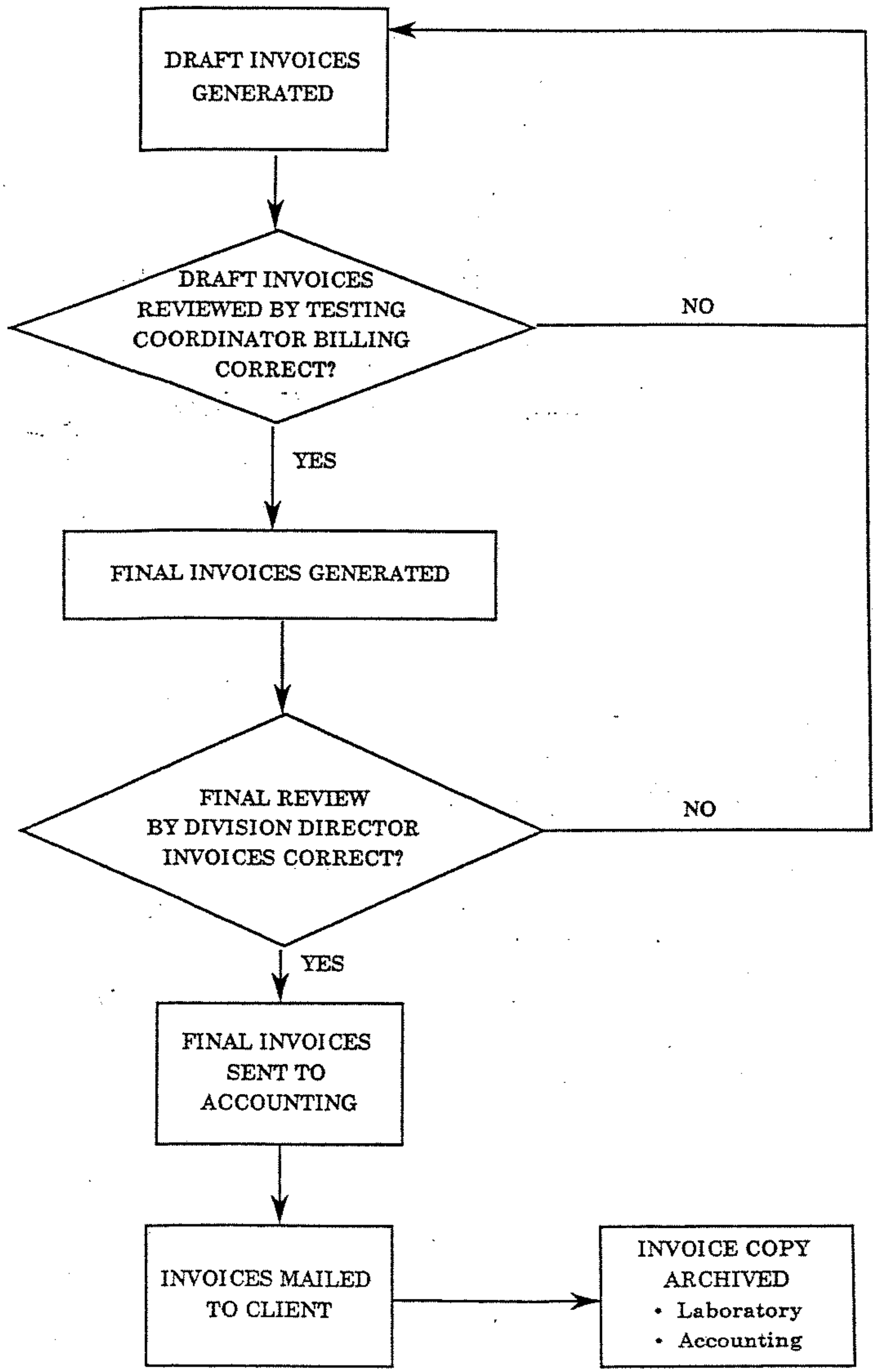
LABORATORY SAMPLE TRACKING SYSTEM
DATA PRODUCTION AND REVIEW



LABORATORY SAMPLE TRACKING SYSTEM
DRAFT AND FINAL REPORT



LABORATORY SAMPLE TRACKING SYSTEM
INVOICING



DISTRIBUTION/TRAINING LOG

LABORATORY SAMPLE RECEIVING,
LOGIN AND STORAGE
STANDARD OPERATING PROCEDURES

SOP NUMBER: **SOP-404**

REVISION NUMBER: **13**

RECEIVED BY/DATE:

W. Schwab WS

F. Rivers FR

~~R. Townsend~~

Signature above signifies acknowledgement of responsibility to know and follow the contents of this document. It also signifies receipt of training covering all new aspects of the SOP.

TRAINED BY:

EFFECTIVE DATE: **06/29/09**

PLEASE COLLECT OLD SOPs AND RETURN WITH SIGNED FORM TO QAO